

AC Engineering/SAIC Joint Annual Progress Report December 10, 1993

1. **Contract Name:** Development and Application of Contamination Technology for MSFC Managed Space Systems
2. **Contract Nr:** NAS8-39244
3. **Reporting period:** December 10, 1992 to December 10, 1993 . .
4. **Technical Progress:** This is the second annual report for this contract. During this time period, studies were concluded which proved that in-process corrosion protection is not required during RSRM case processing. Also completed were a series of tests evaluating the effects of environmental exposure and contamination on 2219-T87 aluminum (Space Shuttle External Tank) OSEE response and bonding properties. Correlations were developed between OSEE response, contamination type/level, and primer adhesion. The results showed that the wet tape and water break free tests currently employed during ET processing may not detect bond affecting levels of some potential contaminants; however, the contaminants were detected with OSEE analysis. Finally, exposure/contamination studies were initiated with HP9-4-30 steel. HP9-4-30 was selected for evaluation because it represents a class of metals common to MSFC managed space flight systems which are less prone to oxidation than D6AC steel or aluminum.

The major accomplishments for this report period were as follows:

1. Completed a 1 year aging study with D6AC steel which had been exposed to 100°F and 60%RH for seven days prior to bonding with NBR insulation.
2. Conducted a study to evaluate the effects of environmental exposure and contamination type/level on 2219-T87 aluminum OSEE response and adhesion to Space Shuttle External Tank primer.

N94-27421

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G3/18 0209778

(NASA-CR-193934) DEVELOPMENT AND
APPLICATION OF CONTAMINATION
TECHNOLOGY FOR MSFC MANAGED SPACE
SYSTEMS Annual Progress Report, 10
Dec. 1992 - 10 Dec. 1993 (AC)
93 p

3. Initiated exposure/contamination tests with HP9-4-30 steel and EPDM insulation.

Effects of Environmental Exposure on D6AC/NBR Insulation Bond Strength

Exposure of unprotected D6AC steel to environmental conditions typically found in the RSRM processing facility results in surface corrosion. The objectives of this effort were to quantify the effects of environmental exposure (temperature/RH/time combinations) on D6AC/NBR (RSRM bondline) adhesion, and to establish whether use of HD-2 grease for in-process corrosion protection could be eliminated without degrading bond strength.

A Taguchi based experiment matrix was employed to evaluate the effects of a broad range of temperature, relative humidity and exposure time combinations on D6AC/NBR adhesion. Results from these studies are shown in Table I (panels E1-E8), and the bond specimen configuration is shown in Figure I. Zero time bonding properties were not affected by the exposure conditions (effort completed during 1992).

To address concerns of bondline aging after exposure, 4 D6AC panels were exposed to 100°F/60%RH for seven days prior to bonding with NBR insulation, then placed in ambient storage (75-80°F, 25-45%RH). Panels E83MB, E83MA and E86MB have been tested after 3, 6 and 12 months of aging, respectively; the results are summarized in Table I. Insulation Shore "A" hardness did not change after one year of aging. The hardness ranged from 62-74 at zero time with unexposed panels (T001-T006), and from 65-73 after 12 months with the exposed panel (E86MB). Peel strength averaged 222 pli after 12 months, which was equivalent to the zero time peel strengths of unexposed panels (197-224 pli). Tensile strength was also unchanged after one year, averaging 719 psi. All of the peel and tensile specimens exhibited 100% cohesive insulation failures.

Based on these results, exposure of unprotected D6AC steel to 100°F/60%RH for time periods up to 1 week (which are the most extreme conditions expected during RSRM case processing) did not affect adhesion to NBR insulation or insulation properties. As a result of this effort, a recommendation was made by the Director of the MSFC Materials and Processes lab that use of HD-2 grease can be eliminated for RSRM case in-process corrosion protection because the oxide formed under typical exposure conditions did not act as a contaminant to the bondline.

2219-T87 Aluminum (External Tank) Exposure/Contamination Study

A study was conducted to quantify the effects of environmental exposure and contamination type/level on the bonding properties and OSEE response of 2219-T87 aluminum. The objectives were to evaluate the OSEE analysis technique as a potential method for contamination detection and quantification during ET processing, and to develop correlations between OSEE signal response, contamination, and ET primer adhesion for a variety of contaminant types which are likely to exist in the ET processing facility.

Approach

Initially, OSEE response versus time was measured for uncontaminated aluminum panels exposed to a range of temperature and relative humidity conditions which the ET is likely to encounter during processing. This provided baseline response trends (due to surface oxidation) that could be expected for an uncontaminated ET in typical manufacturing environments. A Taguchi L8 orthogonal array design (Table II) was selected for this experiment. Aluminum panels (8"x12"x1/8") were cleaned and deoxidized, then exposed to relative humidity levels of 20 or 60%, temperatures of 65 or 100 degrees F, and exposure times of 48 or 96 hours. The 48 hours represented the typical time delay between case cleaning and primer application, and 96 hours was the maximum delay allowed by the processing specification. The RH and temperature extremes were representative of environmental conditions

found at the manufacturing facility. Following exposure to these conditions, the plates were coated with ET primer, which was cured and tested for adhesion to the substrate. Approximate absolute moisture and dew points for the selected environmental conditions are shown in Table III.

In order to gain a better understanding of the effects of environmental conditions on aluminum oxide formation, test panels exposed to the environmental conditions outlined in Table II were examined for aluminum oxide barrier thickness. To measure oxide barrier thickness, a freshly cleaned 3"x6"x1/8" aluminum panel and an exposed panel of the same size were connected to a variable voltage DC power supply and suspended in a 3% tartaric acid solution adjusted to pH 5.5 with ammonium hydroxide. The voltage was gradually increased and was plotted versus current flow. The aluminum oxide barrier thickness in Angstroms was calculated by multiplying 14 times the highest voltage that did not produce a pronounced increase in current flow. The test apparatus is shown in Figure II.

To evaluate the effects of surface contamination, aluminum plates exposed to the environmental conditions outlined in Table II were coated with 1, 5 or 10 mg/ft² of CRC Silicone oil (a model silicone oil) or Kaydol (a model hydrocarbon oil). These materials were representative of the potential contaminants typically found in the ET manufacturing area. The contaminated plates were examined with OSEE, then coated with primer. The cured primer was subjected to adhesion tests to determine the coating level at which the aluminum/primer bond was affected. Comparison of the baseline (uncontaminated) and contaminated panel OSEE responses established the sensitivity of the instrument to the contaminants; i.e., the concentration level at which the OSEE response fell out of the "envelope" of response trends expected as a result of normal surface oxidation. The test matrix, shown in Table IV, combines the Taguchi outer array designs for the two contaminants studied.

Application of Contaminants to Test Panels

Solutions of contaminant (CRC oil or Kaydol) dissolved in methyl chloroform were spray applied to the test panels using a Binks Wren air brush (model 59-10012) pressurized with nitrogen gas to 25-30 psi. Contamination levels

(determined by measuring the weight change of aluminum witness foils sprayed along with the panels) within ± 0.5 mg/ft² of the target levels (1.0, 5.0 or 10.0 mg/ft²) were consistently and reproducibly achieved with this application technique. A summary of the cleaning, priming and testing procedures are shown in Table V.

OSEE Analysis of Uncontaminated 2219-T87 Panels

Tables VI, VII and VIII summarize the results of the core aluminum environmental exposure experiments. The OSEE data shown in Table VI (taken with the environmental chamber system, generation II) showed signal drops of 72-830 cV. The data segregated into 2 groups which were directly related to exposure temperatures; signal drops from 72-230 cV were observed at 65°F, and significantly larger drops from 560-830 cV were seen at 100°F. Figures III and IV show typical OSEE response versus exposure time curves.

The OSEE data in Table VII was obtained with the laboratory environment (generation II) system at room temperature/RH conditions (typically 75-80°F/20-40%RH). Initial OSEE measurements were made immediately before placing the panels in the environmental chamber, and final OSEE measurements were made immediately upon removal from the chamber. Consistent OSEE signals were observed for each panel set after cleaning (values averaging 964-1208 cV), indicating that the zero-time state of the panels was equivalent from Run to Run.

Table VIII shows a comparison of the mean OSEE signal changes for the laboratory (generation II) system versus the response changes observed in the environmental chamber (generation II) system for each experiment. Excluding Run REA1, the lab and chamber OSEE systems exhibited equivalent OSEE signal drops for the experiments performed at 65°F (72-230 cV in the chamber versus 86-239 cV on the lab system). This was probably due to the similarity of the laboratory ambient room conditions and the temperature/RH conditions of Runs 1-4 (65°F/20-60%RH). There was not a correlation between the lab and chamber OSEE data for the 100°F Runs (Runs 5-8). For the purposes of this study, the OSEE response data from the environmental chamber were considered to be more

appropriate since analysis of the ET would likely be performed under a variety of environmental conditions.

OSEE Analysis of Kaydol and Silicone Coated Panels

Following environmental conditioning and OSEE analysis, the panels were coated with approximately 1, 5 or 10 mg/ft² of Kaydol or CRC Silicone oil, reanalyzed for OSEE response, and then coated with ET primer. As shown in Table VII, application of Kaydol or CRC Silicone resulted in further attenuation of the OSEE signal. Application of 1 mg/ft² CRC Silicone resulted in significant signal drops ranging from 200-638 cV. Signal drops were even more pronounced at higher silicone coating levels. The OSEE attenuation with Kaydol at 1 mg/ft² was not always significant, ranging from 30-290 cV. However, at 5 mg/ft² and above the hydrocarbon was always detected, with signal drops ranging from 220-550 cV.

Wet Tape Adhesion Test Results

Results from wet tape adhesion testing of the panels are shown in Table IX. All of the uncoated panels passed the test. At 1 mg/ft² neither Kaydol or CRC Silicone produced ET primer adhesion failure; however, some panels coated with this level of CRC Silicone exhibited "fish-eyes". One panel coated with 5 mg/ft² Kaydol (panel set EA3) and one panel coated with 10 mg/ft² Kaydol (panel set EA2) failed the wet tape test, but the majority passed. With CRC Silicone, primer adhesion failures were seen at 5 and/or 10 mg/ft² for all panel sets except 1, 4 and 6, which passed at all levels. All panels coated with 5 and 10 mg/ft² CRC Silicone exhibited "fish-eyes".

Water Break Free Testing of Aluminum Contaminated With Kaydol or CRC Silicone

Tests were conducted to compare the Kaydol and Silicone coating level at which aluminum failed the water break free test (which is sometimes employed in ET processing to determine tank cleanliness) with the level that could be detected by OSEE analysis.

Aluminum panels cleaned by the procedure shown in Table V were scanned with the OSEE system, then coated with various levels of Kaydol or CRC Silicone. Table X and Figure V summarize the results of the water break free tests. Kaydol induced a water break free test failure between 1 and 2 mg/ft², and CRC Silicone produced failure between 4 and 5 mg/ft². Thus, the technique appeared to be more sensitive to hydrocarbon contaminants than to silicones. Although the OSEE technique detected both contaminants at low levels, the signal attenuation for Kaydol was only 60 cV at 1 mg/ft², which was not considered significant since it was within 2 standard deviations of the mean OSEE response of the uncontaminated panel (1019 cV average). The OSEE signal drop for Kaydol at 2 mg/ft², where water break failure occurred, was significant at 212 cV. The panel coated with CRC Silicone at 1 mg/ft² exhibited a 131 cV drop, which was greater than two standard deviations from the mean of the uncontaminated panel (1076 cV average) and therefore considered to be a significant signal attenuation. Water break free failure occurred at approximately 5 mg/ft² with CRC Silicone, therefore OSEE analysis detected the contaminant at a lower level than could be detected by the water break test. A plot of final OSEE/initial OSEE ratio versus contamination level for the two coatings is shown in Figure V. Silicone had a greater attenuating affect on OSEE response than Kaydol at concentration levels of 1 to 10 mg/ft², but the signals for both were fully attenuated (initial OSEE/final OSEE ratio of 0.2) at levels of 10 mg/ft² and above.

Tensile Adhesion and Pencil Hardness Tests

Because wet tape testing was not considered to be the best indicator of aluminum/primer adhesion, tensile adhesion and pencil hardness measurements were performed on the primed aluminum panels in order to obtain quantitative bond strength data.

Tensile adhesion was measured by bonding 1.25" diameter steel buttons to the primed panels with Versilok 201 (room temperature cure epoxy adhesive), then pulling with an Instron at 0.05"/minute. The results are summarized in Table XI and Figures VI and VII. Tensile strengths of the uncoated panels ranged from 1083 psi (set EA3) to 1683 psi (set REA6); however, the actual primer/metal bond

strength was higher than these averages because the failures occurred predominantly (50-100%) at the secondary bond interface (Versilok/primer). Panels coated with 1, 5 or 10 mg/ft² Kaydol exhibited tensile strengths averaging 988 psi to 1754 psi, and predominantly Versilok/primer failure modes. Thus, this test did not detect any difference in bond strength between uncoated panels and those coated with up to 10 mg/ft² Kaydol. Perhaps a difference would be observed if failures could be forced to the primer/aluminum interface, but even with the undesirable failure modes the bond strengths were acceptable. Panels coated with CRC Silicone exhibited both a drop in tensile strength and a change in failure mode to predominantly primer/metal adhesive failures. Tensile strengths ranged from 500 psi to 1000 psi with 1 mg/ft² CRC Silicone (two exceptions were panel set EA8, which at this level measured 1257 psi, and panel set EA5, which measured 1321 psi), with failure modes averaging 50 - 100% primer/aluminum adhesive failures. Panels exhibited 100% primer/metal adhesive failure and average tensile strengths ranging from 90 psi-316 psi with 5 and 10 mg/ft² CRC Silicone, which were well below those of the clean panels (one exception was panel REA6, which at 10 mg/ft² measured 521 psi, possibly as a result of uneven distribution of silicone across the panel surface).

Pencil hardness tests were also conducted to quantify the effects of exposure/contamination on primer/aluminum adhesion; the results are summarized in Table XII. Tests were performed according to specification ASTM-D-3363 using a set of calibrated drawing leads (A.W. Faber Castell 9000) meeting the following scale of hardness (with hardness increasing from left to right):

6B-5B-4B-3B-2B-B-HB-F-H-2H-3H-4H-5H-6H

The pencil leads were prepared by removing approximately 0.25" of wood from the point of the pencil, leaving an undisturbed, unmarked cylinder of lead. The lead was then held at a 90 degree angle to 400 grit sand paper and rubbed until a flat, smooth cross section was obtained. Panels were tested by holding the pencil at a 45 degree angle to the panel surface, then pushing in approximately 0.25" strokes. Sufficient pressure was applied to either scrape the film from the surface or crumble the pencil lead.

Beginning with the hardest lead (6H) and then using successively softer leads, the procedure was repeated until a pencil was found that did not scrape the film from the surface, which was considered to be the film pencil hardness. As shown in Table XII, the baseline (uncoated) panels and panels coated with up to 10 mg/ft² Kaydol exhibited pencil hardness values of 5H or 6H (except panel set EA3, which was 3H with 10 mg/ft² Kaydol), which are the highest levels on the pencil hardness scale. Thus, as with the tensile adhesion tests, no significant difference was observed between the baseline and Kaydol coated panels. Panels coated with 1 mg/ft² CRC Silicone also measured 5H or 6H on the pencil hardness scale (again with the exception of panel set EA3, which was 3H), which was surprising since these panels exhibited reduced tensile strengths compared to the baseline panels. Panels with 5 or 10 mg/ft² CRC Silicone exhibited significantly lower values of up to 7 pencil hardness units less than the baseline panels.

Based on the results of the pencil hardness and tensile adhesion tests, the presence of Kaydol on aluminum at levels up to 10 mg/ft² did not appear to significantly affect aluminum/ET primer adhesion. The reason for this was unclear, but it is possible that the Kaydol was dissolved into the primer solvent reducer during the primer spraying procedure, and was therefore no longer at the primer/aluminum interface where it could affect the bonding properties of the metal. The aluminum/primer bond was significantly affected at all 3 levels of silicone contamination. However, most of the panels passed the wet tape adhesion test currently used on the space shuttle external tank (Table IX).

Conclusions From Aluminum/Primer Bond Study

Based on the results of the wet tape, tensile and pencil hardness tests of 2219-T87 aluminum panels exposed to the environmental condition extremes of Table II, then coated with 0, 1, 5 or 10 mg/ft² Kaydol or CRC Silicone prior to primer application, neither the oxides formed during environmental exposure or Kaydol up to levels of 10 mg/ft² acted as contaminants to the aluminum/primer bond. The aluminum/primer bond was affected by CRC Silicone at levels of 1 mg/ft² and above; however, tests currently used

during ET processing (wet tape and water break free) were not sensitive to the changes in bond strength occurring with the lower levels of Silicone contamination. Most of the Silicone coated panels passed the wet tape test at levels of 5 mg/ft² and below, and passed water break free testing at 4 mg/ft² and below. The Silicone also did not fluoresce under black light exposure up to levels of 10 mg/ft². OSEE analysis was more sensitive to CRC Silicone on aluminum than the other tests, detecting the contaminant at levels of 1 mg/ft² and above.

Statistical Analysis of Environmental Exposure/Bond Data

Statistical analysis of the environmental chamber OSEE response data was performed to determine the contributions of temperature, relative humidity, exposure time, contamination, and their interactions on the OSEE response and bonding properties of 2219-T87 aluminum. The analysis was performed using Taguchi analysis software provided by Ralph Kissel - EB24.

Statistical analysis of the environmental factors and their interactions are shown in Table XIII, with chamber delta OSEE readings as the response. The responses exhibited a high standard deviation of 277, with exposure temperature having the largest percent contribution to variation at 88%. This was expected based on the obvious trend in the data; at 65°F aluminum exhibited OSEE signal drops ranging from 72-230 cV, while at 100°F the OSEE signal changes were much larger at 560-830 cV. There was not an obvious correlation to exposure humidity or time. Thus, temperature would need to be closely controlled to achieve consistent analysis of 2219-T87 aluminum surfaces.

Table XIV summarizes the results of the factor analysis of primer tensile adhesion values for uncoated (baseline) aluminum panels exposed to the environmental conditions of Table II. Tensile adhesion strengths averaged 1182-1697 psi, with relative humidity contributing 83% to the observed variability. However, it should be noted that the panels exhibited predominantly (50-100%) primer/Versilok adhesive failure, therefore the tensile adhesion values used to perform the calculations did not reflect actual primer/aluminum bond strengths.

Tables XV and XVI summarize the results of OSEE and primer tensile adhesion tests on aluminum panels exposed to the environmental conditions shown in Table II, and then coated with 0, 1, 5 or 10 mg/ft² of Kaydol or CRC Silicone. Factor analysis of the tensile adhesion data for panels coated with Kaydol (Table XVII) showed that relative humidity (39%) and the interaction of temperature/relative humidity/exposure time/contamination (40%) were the predominant contributors to the data variability. However, as with the baseline (uncoated) panels, the Kaydol coated panels exhibited predominantly primer/Versilok adhesive failures (50-100%), so the data used to perform the calculations did not truly reflect the primer/aluminum bond strengths for these specimens. Factor analysis of the CRC Silicone coated panels (Table XVIII) showed that contamination level was the predominant contributor (68%) to the variability in tensile adhesion strength. This was not unexpected based on the significant reduction in tensile strength observed at even low levels of silicone contamination. Tables XIX and XX summarize the factor analysis results with delta OSEE as the response for Kaydol and CRC Silicone, respectively. As was expected, contamination level was the largest contributor to OSEE response variability at 90% for Kaydol and 76% for CRC Silicone. Thus, contamination level had a more pronounced effect on OSEE response than environmental exposure conditions.

Barrier Oxide Thickness Measurement

Results of the barrier oxide thickness measurements are shown in Table XXI.

The Al₂O₃ thicknesses ranged from 9-19 Å, with no obvious correlation to exposure conditions. It was expected that panels exposed to the higher temperature (100°F) would have a slightly higher Al₂O₃ thickness, and this would seem to be supported by the greater OSEE signal attenuations observed at 100°F (Table VI). However, the test did not detect a difference in barrier oxide thickness between the two exposure temperatures. Although the barrier oxide measurements were performed as quickly as possible after removing panels from the environmental chamber, the panels would immediately begin to equilibrate to the room temperature, which would affect Al₂O₃ thickness. This may

explain why the panel sets exposed to 100°F exhibited similar barrier oxide thicknesses to panels exposed to 65°F.

HP9-4-30 Environmental Exposure/Contamination Study

The objectives of this effort were to evaluate the effects of environmental exposure and contamination type/level on the OSEE response and bonding properties of HP9-4-30 steel. Test panel processing methods and environmental exposure limits were selected based on conditions the ASRM case would likely experience at the Yellow Creek facility in Iuka, Mississippi. While the results from this study would be directly applicable to ASRM case processing, they are also useful because HP9-4-30 steel is a good model for materials which are less prone to oxidation than D6AC steel or aluminum.

Approach

The test matrix, a Taguchi L8 orthogonal array (Tables XXII and XXIII), was based on processing flow information provided by Aerojet personnel. The ASRM production facility was designed so that case grit blasting and aqueous cleaning could be performed in a temperature controlled environment ($75 \pm 5^\circ\text{F}$), and the remaining case processing (inspection, Chemlok application, etc.) could be performed in a temperature ($75 \pm 5^\circ\text{F}$) and relative humidity controlled ($\leq 55\%$) environment. The exposure time and RH parameters shown in Table XXII represented the likely range of conditions the ASRM case would experience between completion of the aqueous cleaning process and movement into the airlock (temp/RH controlled) facility. Following the initial exposure of 4 or 48 hours at 40% or 75%RH, test panels were subjected to an additional 24 hours at 55% RH to simulate the likely time delay between arrival in the airlock facility and Chemlok application; during this time the case would be undergoing inspection and preparation for primer application.

After environmental exposure was completed, test panels (8" x 12" x 1/8") were coated with CRC Silicone, Conoco HD-2 grease, or Kaydol, which represented the types of contaminants commonly found in rocket motor processing facilities. The coatings were spray applied to the panels

using a Graco air brush (Model G1265, series B) pressurized to 40-50 psi with nitrogen. Coating level was determined by measuring the weight change of aluminum witness foils sprayed along with the panels. Target contamination levels were 25 mg/ft² and 200 mg/ft² for Kaydol and HD-2, and 2 mg/ft² and 20 mg/ft² for CRC Silicone. The panels were then analyzed for OSEE response (generation II OSEE system), and bonded to EPDM insulation, specification 44010B. Figure VIII shows the bond specimen configuration, Table XXIV describes bond sample preparation, Table XXV shows the process flow, and Figure IX shows the insulation vulcanization conditions.

HP9-4-30 Discoloration During Turco 3878 LF-NC Cleaning

Initially, based on recommendations by Aerojet personnel, Turco 3878 LF-NC aqueous cleaner (20% concentration level, 2 hr. immersion time, 140°F, 4% by volume agitation rate per minute) was used to clean HP9-4-30 steel panels prior to environmental exposure. Several practice cleaning runs with 2-4 panels were successfully completed in a 60-gallon tank, then a set of seven panels was cleaned without problems. However, subsequent attempts to clean panels were unsuccessful due to discoloring of the panel surfaces during immersion in the Turco bath. A significant amount of time was spent trying to understand and remedy the problem, but to no avail. Following is a summary of the observations surrounding this phenomenon.

Two practice cleaning runs were completed without complications in a 60 gallon Turco tank in building 4760. Following the practice runs, a set of seven panels (which were vapor degreased by NAS prior to the Turco cleaning procedure) was successfully cleaned in the same solution. A second set of 7 panels (not vapor degreased) was then successfully cleaned in the tank, but a power outage interrupted the controlled environmental exposure of the panels. The set was then recleaned with Turco, but were charcoal colored when removed from the bath. It was suspected that the bath water level was low (and therefore the cleaner concentration too high), so DI water was added to the tank to bring it to the full line. The discolored panels were grit blasted to remove the residue, then re-immersed in the bath. Once again they discolored. The 60 gallon

tank was then drained, cleaned, and recharged with fresh Turco. The same set of panels was blasted/cleaned, and again discolored. Two previously unused panels were then grit blasted and immersed in the solution, and they also discolored.

At this point, experiments were initiated using a 5-gallon tank in building 4711 and Turco from a different drum than had been used to prepare the bath in building 4760. Utilizing the new Turco solution, one previously unused panel was successfully cleaned. A second panel, which had been blackened and then grit blasted, was then immersed, and came out discolored. The panel cleaned prior to this (which did not discolor) was then re-immersed, and this time it too discolored.

All of the HP9-4-30 panels used for the study were machined at MSFC to obtain a level surface suitable for OSEE analysis, and it was suspected that residual machine cutting oil might be poisoning the Turco solution. UV spectra obtained on various "poisoned" Turco solutions (solutions from which discolored panels were removed) and mixtures purposely spiked with cutting oil seemed to support this theory (Figure X); both exhibited the same shifts in absorbance. To test this, two HP9-4-30 panels provided by Aerojet (which had not been machined at MSFC) were first cycled through fresh Turco solution and did not discolor. Next, two previously unused but vapor degreased ACE panels were processed through the solution and also came out clean. Then two non-vapor degreased ACE panels (which would still contain residual cutting oil) were immersed; they also came out clean. Thinking that perhaps a higher concentration of cutting oil had to be present in the Turco solution before discoloration occurred, the bath was then spiked with up to 4% of the oil. However, panels immersed in these purposely contaminated solutions did not turn black.

A second theory was that residue from the panel heat treating process was tainting the Turco baths. Therefore, two panels which had not been machined, grit blasted or vapor degreased (and would still have residue from the heating process) were successively immersed. Neither panel discolored.

The next experiment was performed to determine if only the ACE steel panels would discolor (Aerojet personnel also observed panel discoloration during Turco cleaning operations, but attributed the phenomenon to elevated bath temperatures). A previously blackened ACE panel was meticulously grit blasted and processed through a known good Turco solution. This panel again became discolored. An Aerojet panel was then immersed and came out clean. A second ACE panel (previously blackened, then grit blasted) was processed through the solution and it discolored. The same Aerojet panel was again immersed, and this time it too discolored.

Based on the experiments described above, it was concluded that discoloration was not exclusive to one container of concentrated Turco 3878 LF-NC, or to one cleaning tank. Discoloration was also not due to a temperature control problem, or to residual cutting oil from the machining operations. Finally, discoloration occurred not only with the ACE panels, but with HP9-4-30 from other sources as well.

Analysis of Turco Solutions

As the experiments described above were progressing, samples of good and "poisoned" Turco solutions were collected for analysis. Table XXVI summarizes the results of pH and conductivity tests performed on the samples. There was not a correlation between pH or conductivity and the propensity of the solution to cause discoloring. All solutions exhibited pH values from 8.5-9.0, and the conductivities ranged from 3.7-12.1 mS. Although there was some difference in conductivity between solutions prepared from a 55 gallon drum of concentrate (9.9-12.1 mS) and those prepared from a 25 gallon drum (3.7-4.3 mS), this did not directly relate to panel discoloration.

Samples of unused and "poisoned" Turco were analyzed by Southeastern Analytical Services (SEAS) to compare the percentages of metals present; the results are shown in Table XXVII. The most significant difference between the fresh and tainted Turco solutions was the concentration of iron. The fresh solution contained less than 0.02 mg/L, and the tainted solution contained 1.09 mg/L. However, it was not clear how this could contribute to the discoloring. Turco

Inc. representatives were provided samples of the solutions and panels for evaluation, but were unable to duplicate the discoloring phenomenon under the conditions used at MSFC and Yellow Creek. However, they did confirm the higher percentage of iron in the "poisoned" solutions.

HP9-4-30 Summary

The steel used to prepare the panels was purchased by Republic Engineered Steel from Air Melt Heat as two billets weighing approximately 4040 lb each. The two billets were heat treated simultaneously by H&H Heat Treating, Inc., heat number 3844507 (Table XXVIII); this heat treatment readied the steel for machining. The panels should have been heat treated per ASRM spec 45000 after machining, but this was not done. Target hardness level for the panels was 450-470 BHN, but the ACE panels averaged only 344 BHN (Table XXIX). However, this alone was not believed to be the cause of the discoloration problem, because some ACE panels did not blacken after repeated cleaning in Turco. The panels were later heat treated to 455 BHN hardness but still discolored, eliminating incomplete heat treating as the cause.

X-ray and ICP analyses showed the panels to be of the proper metallurgy (Table XXX).

Residue Analysis

A blackened panel flushed with Freon 113 had an NVR of 11.8 mg/ft². Microscopic examination of the residue revealed it to be primarily a grit with green and black particles. About 20% of the particles were magnetic. Based on this analysis, possible makeup of the residue included Fe₃O₄, which is black in color and magnetic, and nickel oxides, which are also black. The green particles were possibly complex phosphates, but no phosphate color information could be found. Ellipsometry measurements supported the existence of Fe₃O₄; a layer of approximately 500Å thickness was identified on a blackened panel with optical constants matching those of iron oxide (Figure XI). A second layer of 1000 angstroms was observed on top of the iron oxide, but the composition of this layer was not determined.

Discolored Panel Analysis

Scanning Electron Microscope/EDAX analysis of clean and discolored panels (Figures XII, XIII and XIV, spectra normalized on the Fe peak) revealed a significantly increased signal for oxygen on the discolored panel, which was consistent with the presence of metal oxides. The discolored panels also exhibited higher signals for sulfur, chlorine, carbon and chromium (or, a decreased signal for Fe). Zirconium and silicon were observed on both the clean and discolored panels, and were believed to be due to residual grit blast media (Zircon). No phosphorous was observed, which would exclude the presence of phosphates.

Effect of Discoloration on HP9-4-30 Steel Bonding Properties

One question of interest regarding the discoloration was whether the residue acted as a contaminant to the bondline. To answer this question, bond specimens were prepared using EPDM insulation or EA934.NA epoxy adhesive on panels which were clean (did not discolor after repeated immersions in Turco), discolored, or which had discolored and then been grit blasted to remove the discoloration. Tests were performed with the discolored/blasted panels because if the ASRM case were to discolor during processing, it would be important to know whether the case could continue through normal processing following grit blast removal of the discoloration. Specimens were tested with EA934.NA adhesive because it was believed that the epoxy/steel bond would be more sensitive than the insulation/steel bond to differences in steel bonding properties. Specimens containing EPDM simulated the ASRM case/insulation interface. Results are summarized in Table XXXI.

The clean, discolored, and discolored/blasted panels containing EPDM insulation exhibited similar peel and tensile adhesive strengths. Peel strengths averaged 138-166 lbs max. peel load with 100% failures along the insulation/scrim interface. Tensile strengths averaged 449-489 psi with 100% cohesive insulation failures. Thus, the discoloration did not affect HP9-4-30/EPDM adhesive strength, and was therefore not considered to be a

contaminant to this interface.

Panels bonded with EA934.NA epoxy did exhibit a difference in tensile adhesion (peel adhesion tests were not performed on this interface), but showed that the discolored surface was better than the clean surface for bonding to the epoxy. The clean panels exhibited tensile strengths averaging 225 psi with 80% epoxy/metal failure modes, while the discolored panels averaged 677 psi with only 50% metal/epoxy failure (and 50% cohesive epoxy failures). The best results were observed with the discolored/blasted panels, which averaged 1023 psi tensile adhesion with 100% cohesive epoxy failures. Thus, as with the steel/insulation bond, the discoloration was not a contaminant to the steel/EA934.NA bond.

Evaluation of Brulin 815 GD Aqueous Cleaner

Due to the problems associated with use of Turco 3878 LF-NC, Aerojet initiated studies with alternative aqueous cleaners. Brulin 815 GD was considered to be the top candidate, and did not discolor HP9-4-30 panels which had consistently discolored in Turco 3878 LF-NC. Therefore the decision was made to use Brulin to complete the HP9-4-30 exposure/contamination study.

OSEE Analysis Results

Five sets of seven HP9-4-30 panels have been cleaned in Brulin 815 GD without incident; these panels were used to complete exposure runs 1, 2, 3, 4, and 6 of the current test matrix (Tables XXII and XXIII). Results from the experiments are shown in Table XXXII and Figures XV, XVI. Table XXXII summarizes the OSEE results as measured on the Table I (lab environment, generation II) system. The initial OSEE responses, taken immediately after cleaning in Brulin, were fairly consistent with averages of 476-577 cV for the 5 sets of panels. Post exposure OSEE readings were also similar, with averages ranging from 462-523 cV. Modest signal drops (post exposure minus initial OSEE readings) of 25-61 cV were observed for the range of exposure conditions. The one exception to this was Run 3, which exhibited a positive 47 cV signal change after exposure; the increase was due to adjustments made to the OSEE system between the initial and post exposure measurements, which increased

the sensitivity of the instrument (the Ni standard measured 908 cV when the initial measurements were made, and 980 cV when post exposure measurements were taken).

Table XXXII also shows the changes in OSEE responses resulting from application of HD-2, Kaydol or CRC Silicone grease. CRC Silicone had the most pronounced effect on signal response; signal drops averaging 137-243 cV were observed with 2-7 mg/ft² coating levels, and signals were reduced by 397-483 cV with 15-17 mg/ft² coatings. Kaydol produced signal reductions of 276-508 cV at 20-29 mg/ft², and attenuated the signal by 470-505 cV at 170 mg/ft² and above. Panels coated with 195-250 mg/ft² HD-2 grease exhibited OSEE signals averaging 153-182 cV, which were equivalent to the signal responses of panels coated with 17-25 mg/ft² HD-2 (131-212 cV); this was not unexpected since HD-2 is a photoemitter and exhibits an OSEE response at high coating levels.

Figures XV and XVI show typical plots of OSEE response versus time for the completed exposure cycles (taken in environmental chamber during exposure, OSEE system generation II). For the Runs completed to date, the overall OSEE response changes have been modest, averaging 25-95 cV. For Run 2 (Figure XV) the overall response change was 95 cV (455 cV initial to 360 cV final), and an increase in response of 30 cV was observed when the temperature and RH were changed from 70F/40% to 75F/55% (at 2880 minutes). Panels exposed to the environmental conditions of Run 4 (Figure XVI) exhibited a signal drop of 35 cV during exposure, but did not show a change in OSEE signal when the temperature and RH were adjusted.

Bond Study Results

Following environmental exposure and contamination, panels were bonded to EPDM 44010B insulation for peel and tensile adhesion testing. The results are summarized in Table XXXIII. Shore "A" hardness values after insulation vulcanization averaged 76-87 (± 5), indicating that complete cures were achieved. Uncoated panels from Runs 2, 3, 4 and 6 exhibited peel strengths averaging 103-110 pli and tensile strengths averaging 349-465 psi, with 100% cohesive insulation failures. Inexplicably, the uncoated panel from Run 1 had significantly lower peel strengths

averaging 42 pli (100% insulation failures). Several other panels in the Run 1 set (HP23 with 170 mg/ft² Kaydol, and HP20 with 15 mg/ft² CRC Silicone) also had significantly lower peel strengths than their counterparts from other Runs. The bond specimens for Runs 1 and 2 were vulcanized simultaneously, and since erratic peel values were not observed for the Run 2 panels, the cure cycle must have been adequate (and, as noted earlier, Shore "A" hardness measurements indicated that the insulation was fully cured). Also, since the exposure conditions for Run 1 were less extreme than those of the other Runs, it was unlikely that the lower peel strengths for Run 1 were due to a more significant buildup of oxidation products. Finally, insulation mechanical properties were measured and found to be within specification requirements (discussed later in this report). The cause of the low peel strengths for Run 1 was not obvious, but the data were considered anomalous based on the results from Runs 2, 3, 4 and 6.

Panels containing 17-27 mg/ft² HD-2 grease, 20-29 mg/ft² Kaydol, 180-218 mg/ft² Kaydol, 2-7 mg/ft² CRC Silicone, or 15-17 mg/ft² CRC Silicone were not affected by the coatings; bond strengths and failure modes were comparable to those observed with the uncoated samples. Several panels with these coating types/levels (HP16-Run 3 with 218 mg/ft² Kaydol, HP27-Run 3 with 17 mg/ft² Silicone, HP5-Run 4 with 195 mg/ft² Kaydol) exhibited slightly lower peel strengths (84-87 pli) than the baseline specimens (103-110 pli), but were considered equivalent to the uncoated panels since they failed cohesively in the insulation. However, there was bond strength degradation with HD-2 grease at 210-250 mg/ft². The panel from Run 3 (HP7 with 210 mg/ft² HD-2) had tensile values (459 psi) equal to the baseline panel (463 psi), but exhibited 50% Chemlok to steel adhesive failures. Also, peel specimens from panel sets 1 (HP27 with 250 mg/ft² HD-2), 2 (HP1 with 220 mg/ft² HD-2) and 6 (HP23 with 210 mg/ft² HD-2) showed approximately 5% Chemlok/EPDM adhesive failures, and peels from panel set 4 (HP4 with 195 mg/ft² HD-2) had significantly lower peel strengths (65 pli) than the baseline panel (110 pli).

EPDM 44010 Mechanical Property Tests

Due to the unusual peel strengths observed for panels exposed to test matrix Run 1 conditions, EPDM mechanical properties were tested to see if the insulation had degraded during storage. Table XXXIV summarizes the vendor test results (RM Engineered Products), as well as results from two series of tests after 2 and 6 months of freezer storage at MSFC. Insulation for the most recent tests (10/31/93) was vulcanized along with panels from Run 4 of the current test matrix.

Shore "A" hardness measurements at RM averaged 92, while both series of tests at MSFC averaged 82; all values were above the minimum specification requirement of ≥ 80 . Tensile strength (avg. stress at max load) parallel to the fiber direction was 2457 psi initially (RM data), and at 2188 psi after 6 months of freezer storage was still well above the ≥ 1000 psi requirement. Elongation (avg. max. percent strain) parallel to the fiber direction was also acceptable after 6 months; the criteria for elongation is $\geq 5\%$, and the most recent tests averaged 19%. Mechanical properties perpendicular to the fiber direction (tensile strength = 884 psi, elongation = 78%) were also significantly above the stress (≥ 500 psi) and elongation ($\geq 5\%$) requirements. Thus, the insulation mechanical properties were acceptable and did not appear to have been the cause of the erratic peel values observed for panels from Run 1 of the test matrix.

5. *Issues And Problems:* None

6. *Plans for next reporting period:*

- A. Complete HP9-4-30 test matrix and conduct statistical analysis of results.
- B. Initiate evaluation of UVF analysis system.

FIGURE I
NBR INSULATION / D6AC STEEL
BOND SAMPLE CONFIGURATION

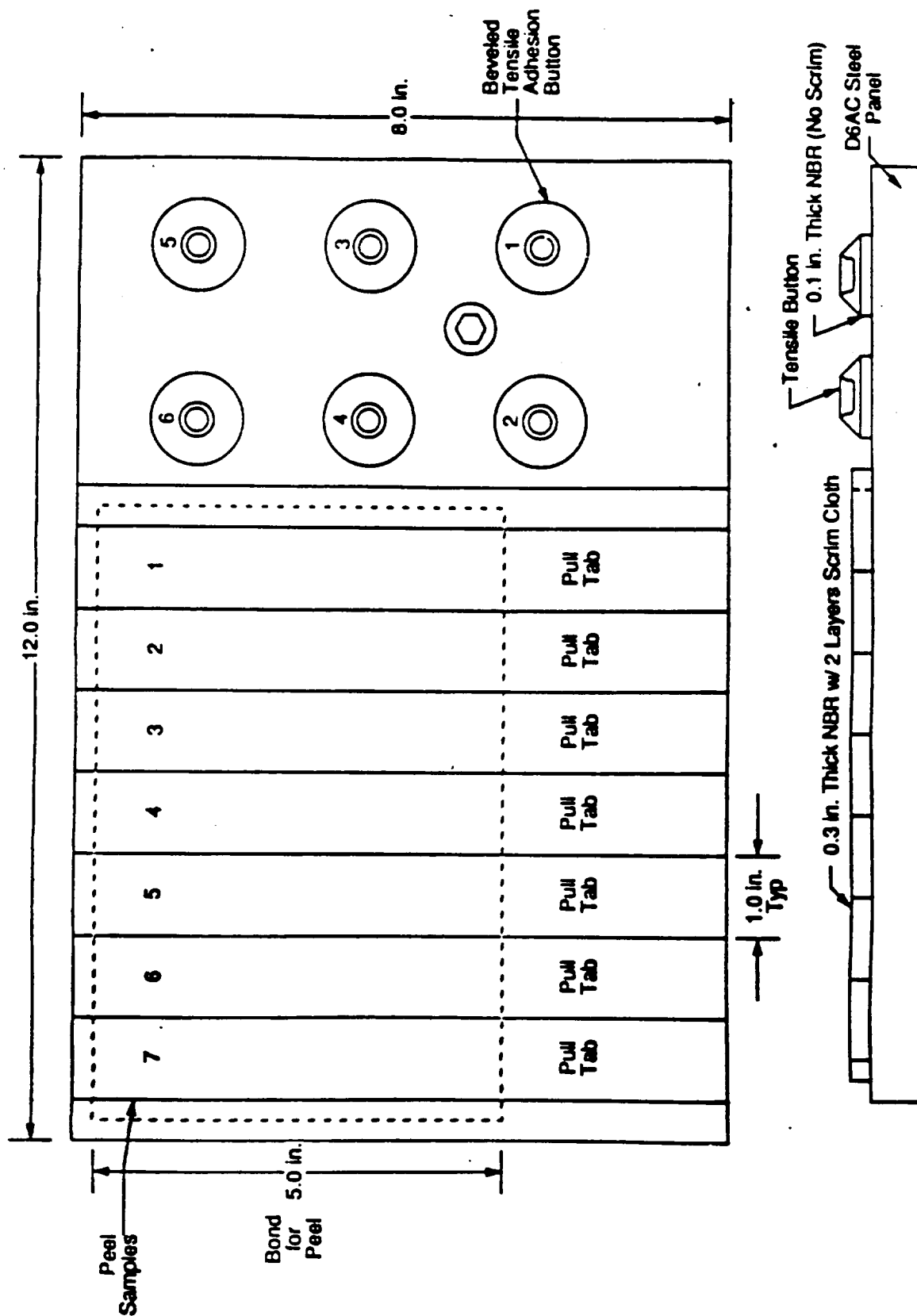
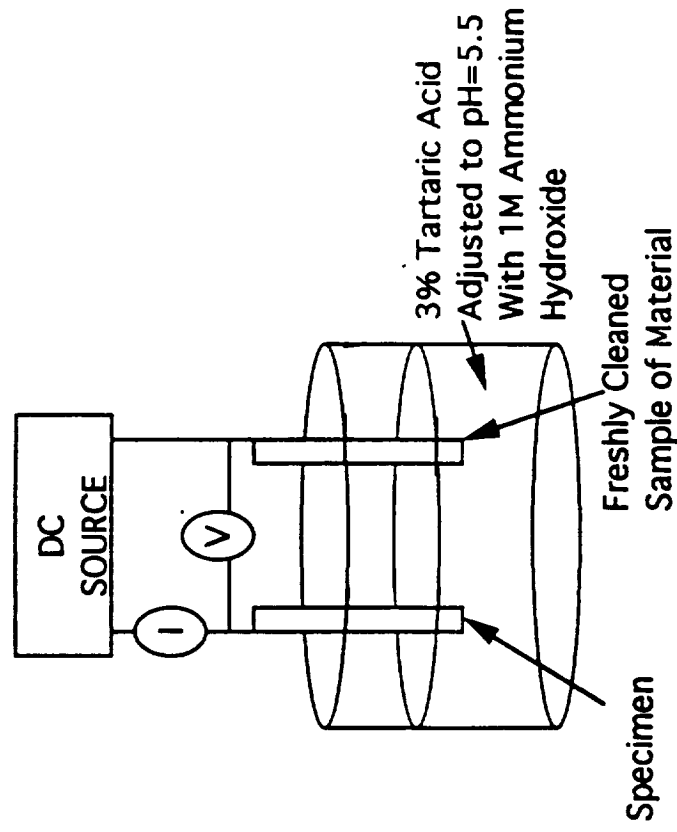


FIGURE II
ALUMINUM BARRIER OXIDE THICKNESS
MEASUREMENT TECHNIQUE



-Increase voltage gradually until I flow above leakage value is obtained

-Once appreciable I starts to flow, small voltage change results in large I increase

-Thickness of barrier in angstroms approx. equal to 14x highest V that does not produce pronounced I increase

FIGURE III: 2219-T87 ALUMINUM
PANEL EA8: 96HRS AT 100F/60%RH

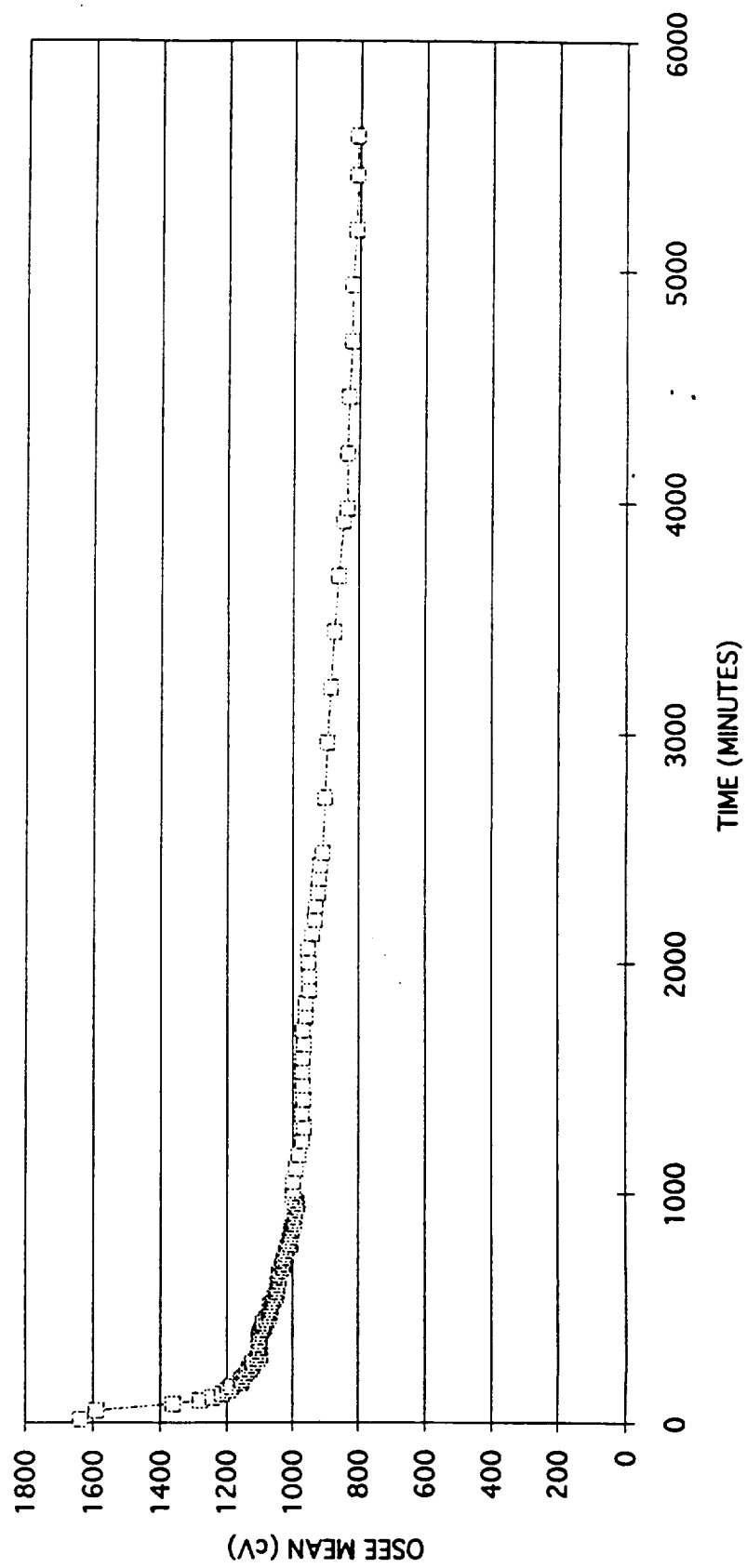


FIGURE IV: 2219 AL PANEL EA2
96HRS AT 65F/20%RH

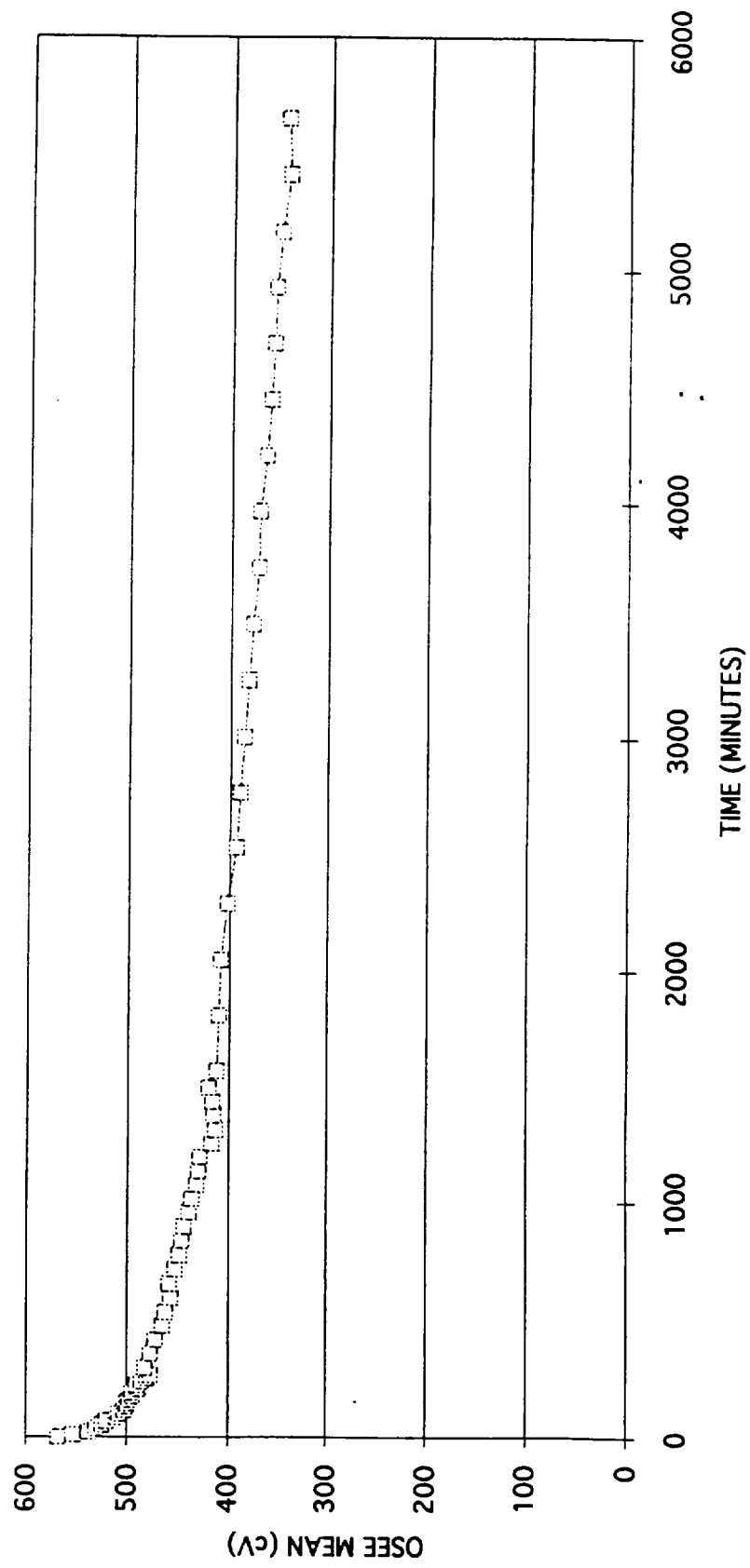


FIGURE V: 2219-T87 ALUMINUM, DELTA OSEE WITH CONTAMINATION

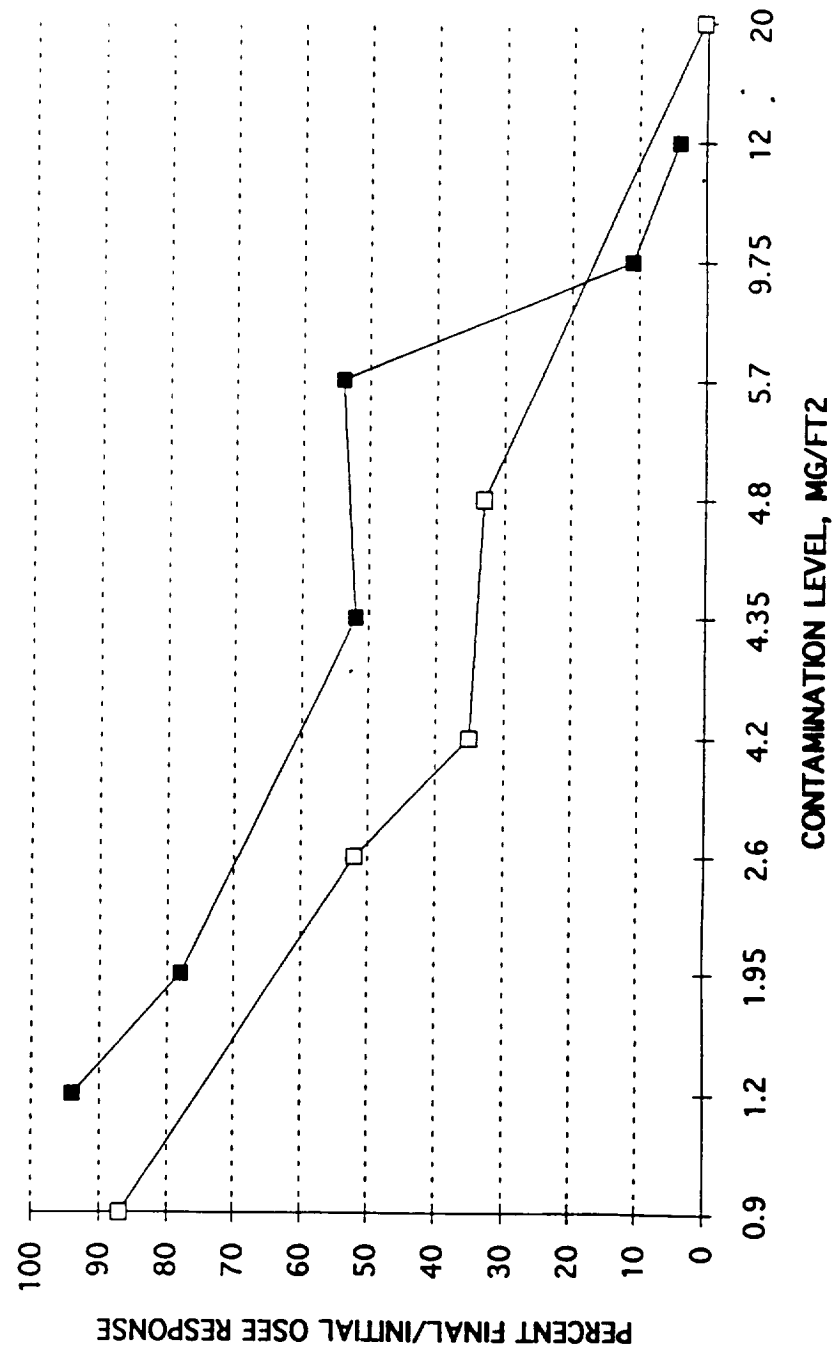
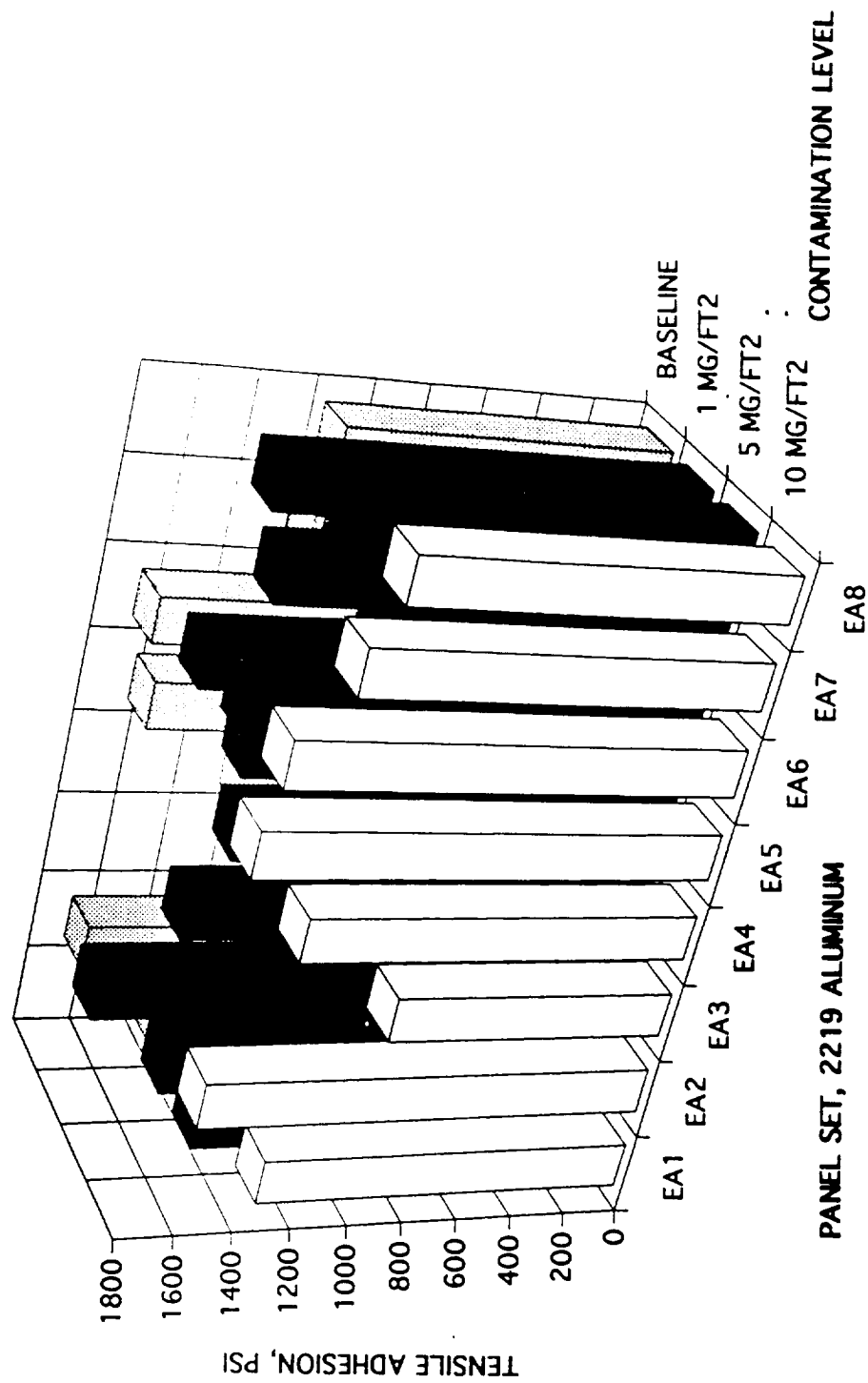
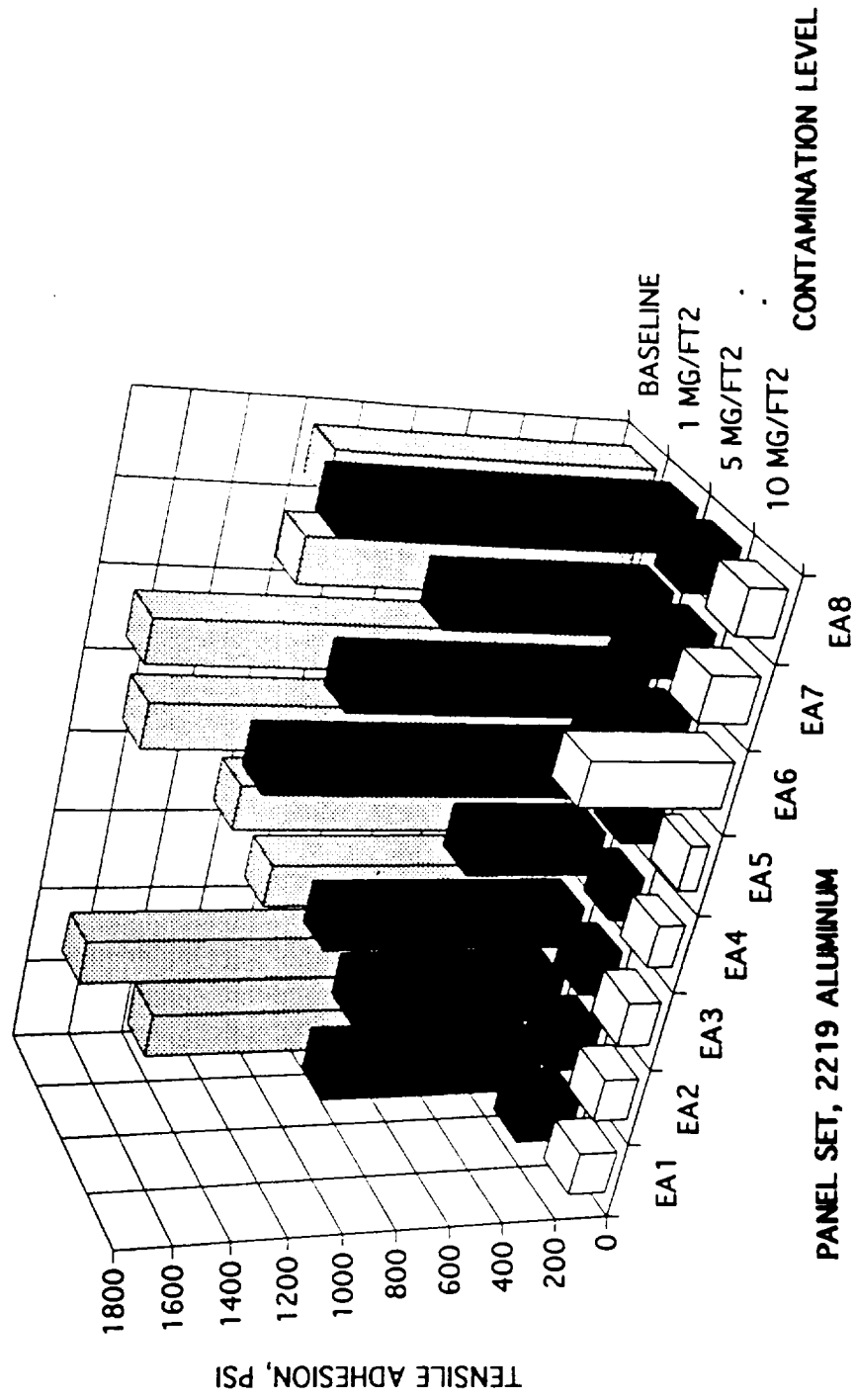


FIGURE VI: PRIMER TENSILE ADHESION VS CONTAMINATION LEVEL, KAYDOL-2219-T87 ALUMINUM ENVIRONMENTAL EXPOSURE/CONTAMINATION STUDY



1.25" diameter tensile buttons bonded to primed aluminum using Versilok 201, then pulled with an Instron at 0.05"/min. All specimens exhibited primer/Versilok failure. AC11a/5/93

FIGURE VII: PRIMER TENSILE ADHESION VS CONTAMINATION LEVEL, CRC
SILICONE: 2219-T87 ALUMINUM ENVIRONMENTAL EXPOSURE/CONTAMINATION
STUDY



1.25" diameter tensile buttons bonded to primed aluminum using Versilok 201, then pulled with an Instron at 0.05"/min. Silicone coated specimens exhibited primer/aluminum failure. AC11b/5/93

FIGURE VIII
EPDM INSULATION/HP9-4-30 STEEL
BOND SAMPLE CONFIGURATION

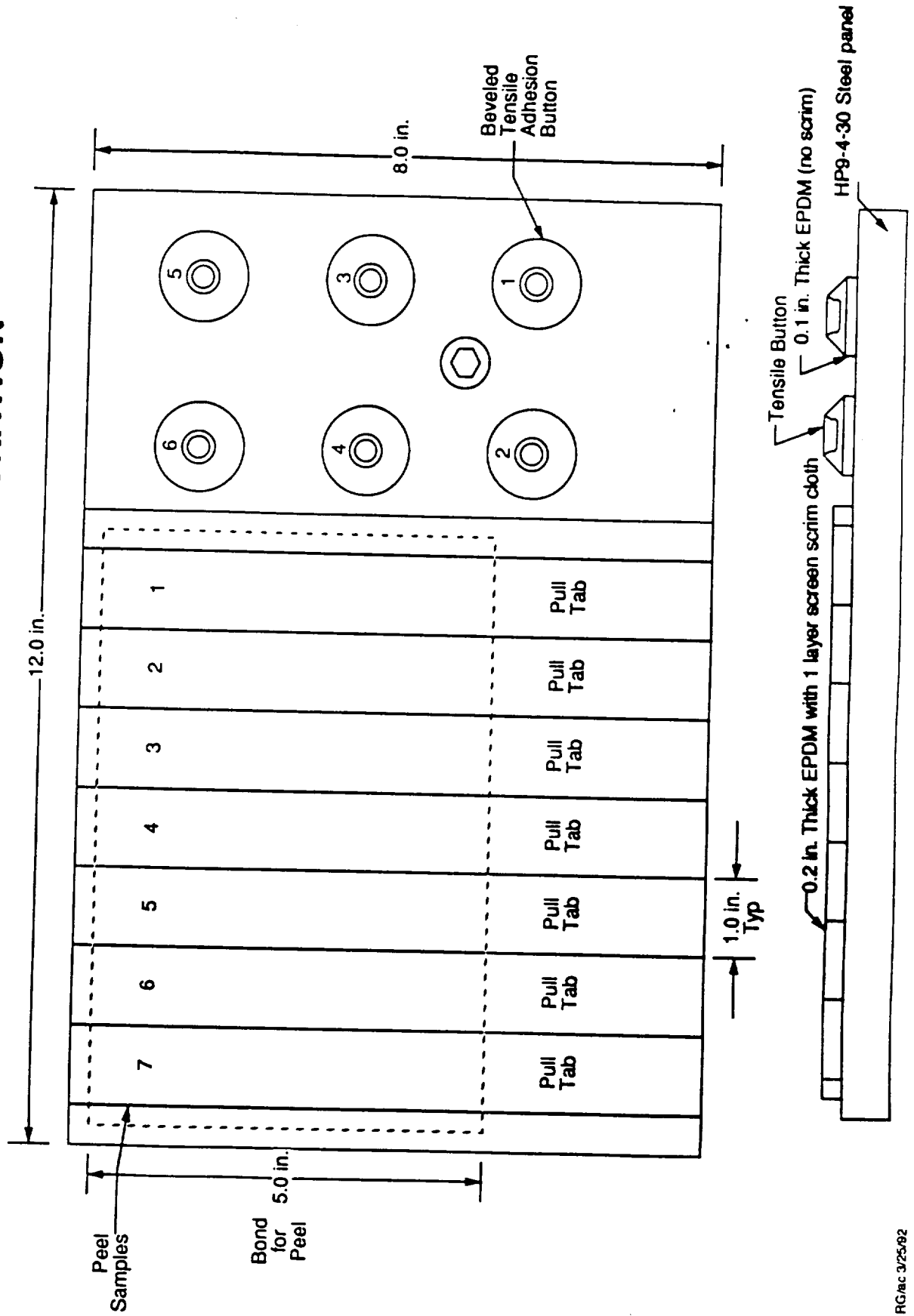


FIGURE IX
EPDM 44010 CURE CYCLE

<u>STEP NUMBER</u>	<u>TEMPERATURE, °F</u>	<u>PRESSURE, PSI</u>	<u>TIME, MINUTES</u>
1	75	0	0
2	85	100	10
3	210	100	25
4	210	100	60
5	320	100	55
6	320	100	120
7	120	100	40
8	85	0	5

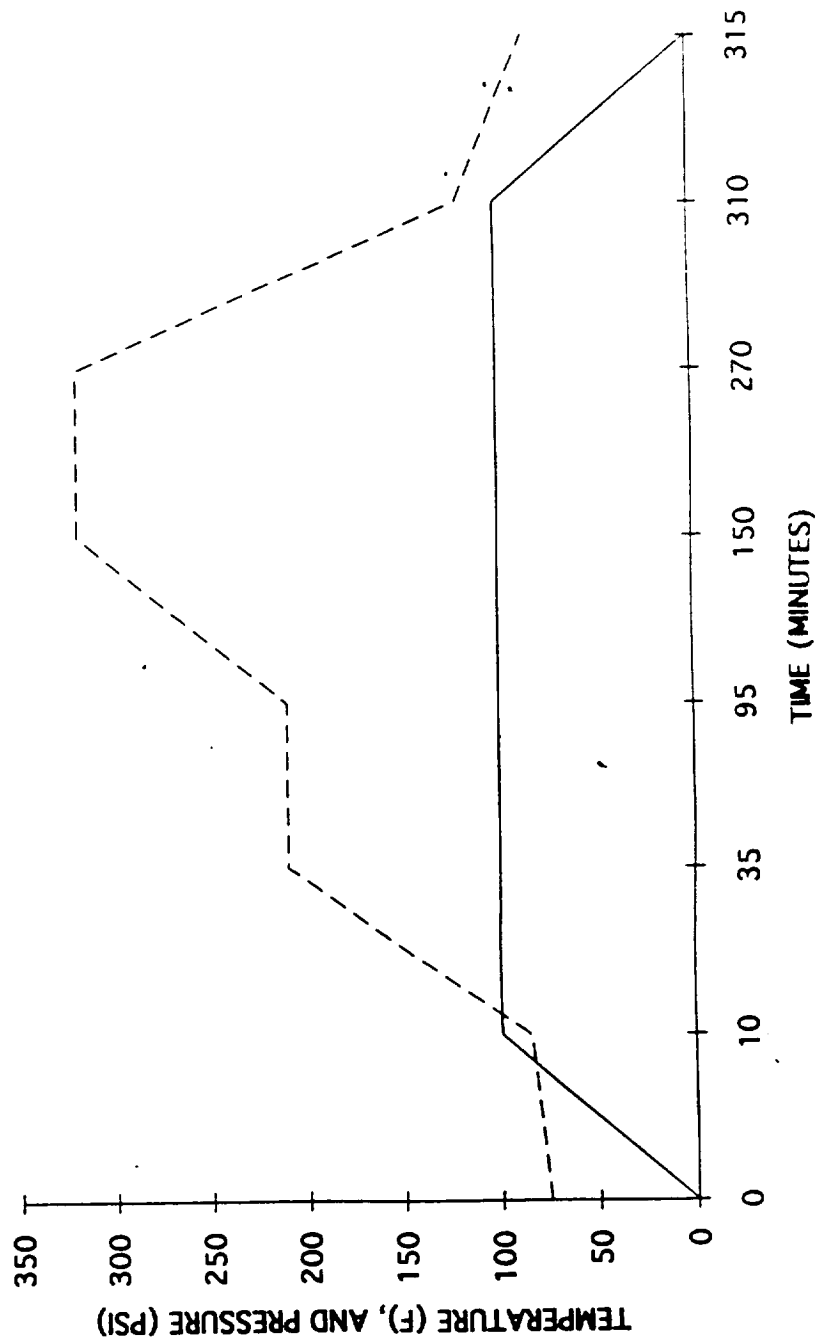


FIGURE X
UV ANALYSIS
FRESH SOLUTIONS VS. SOLUTIONS SPIKED W/
MACHINE CUTTING OIL

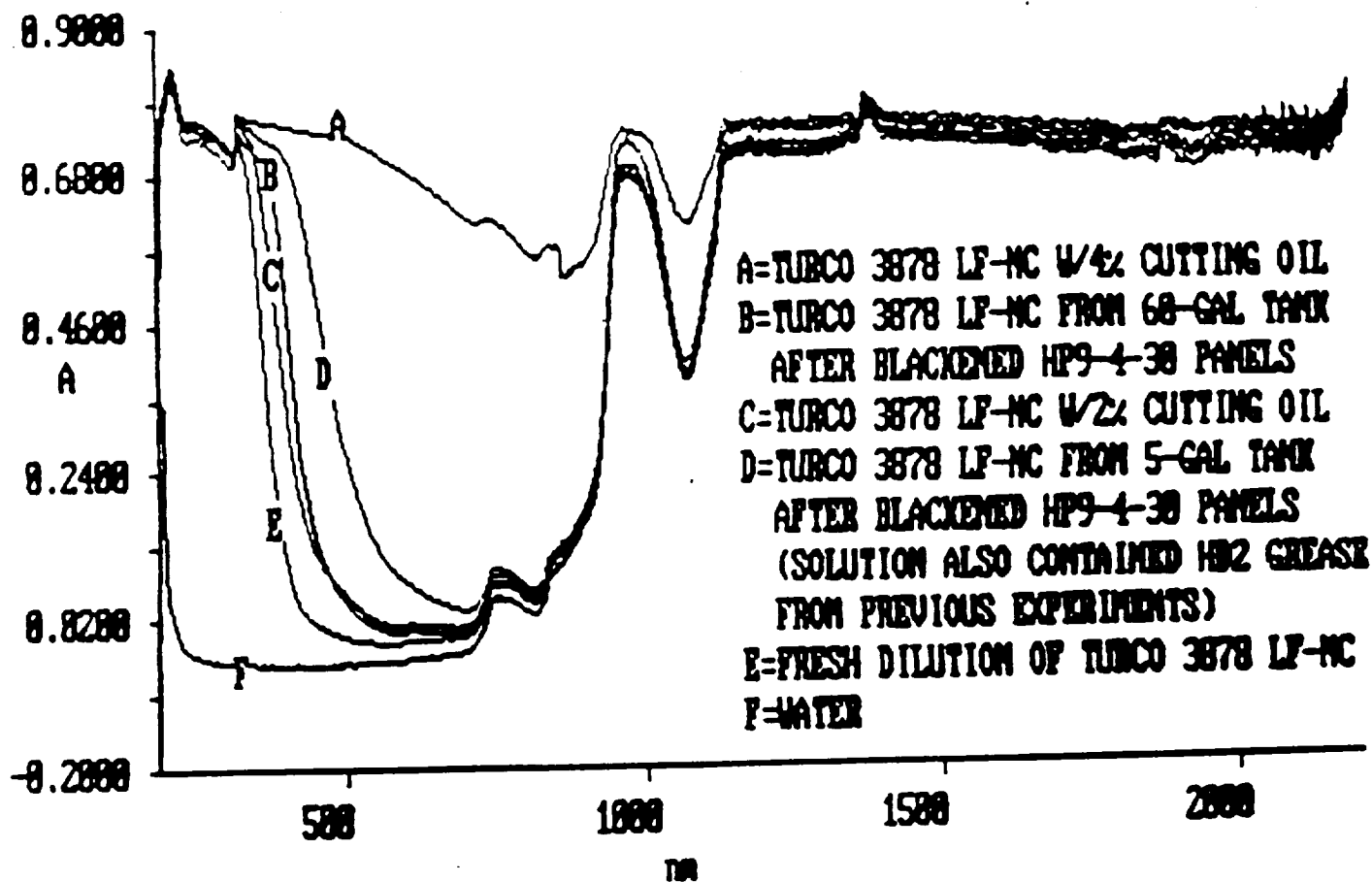


FIGURE XI
ELLIPSOMETER ANALYSIS OF
DISCOLORED HP9-4-30 PANEL
 (LAYER NEXT TO METAL SURFACE MATCHES Fe_3O_4)

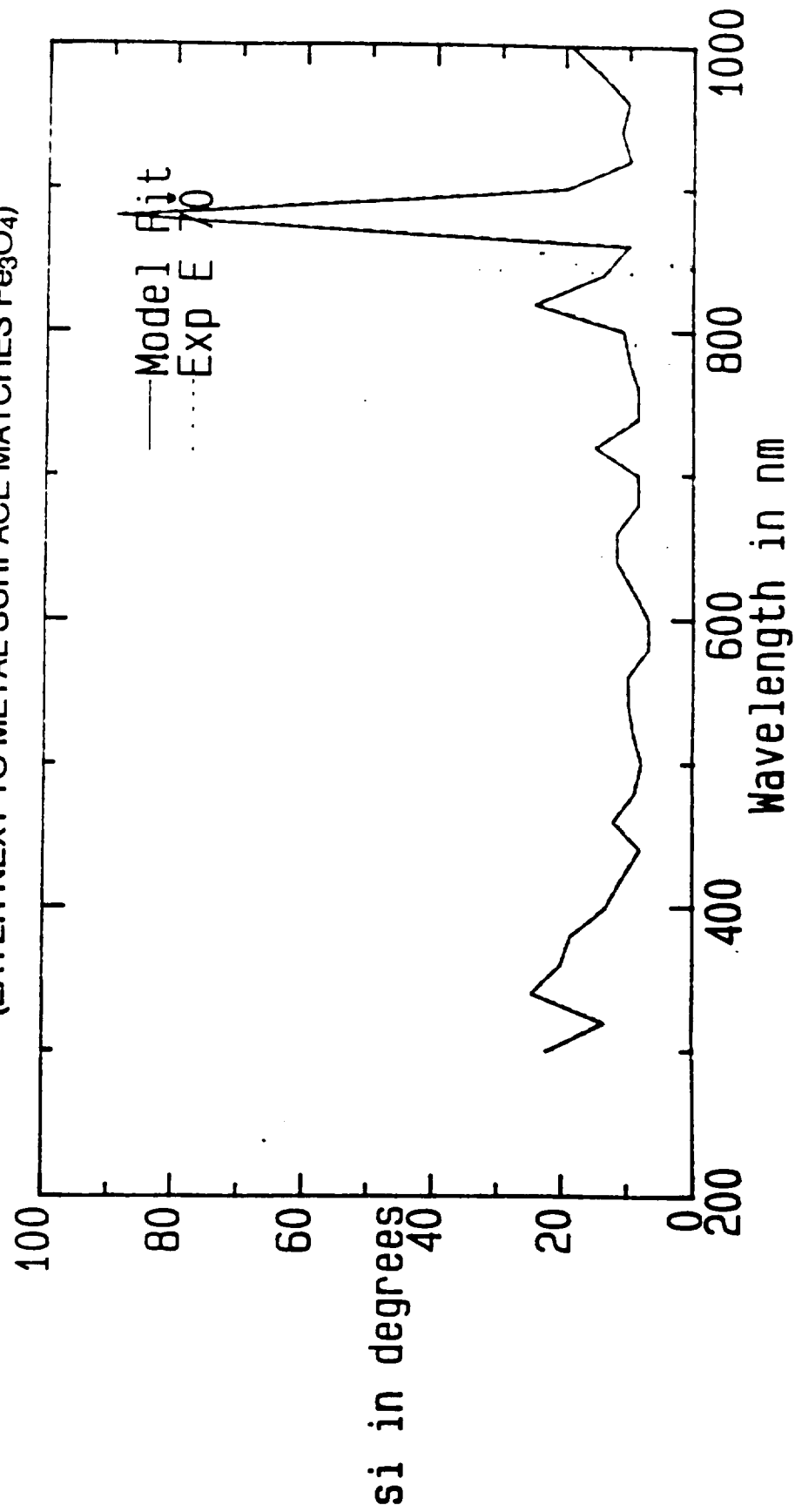
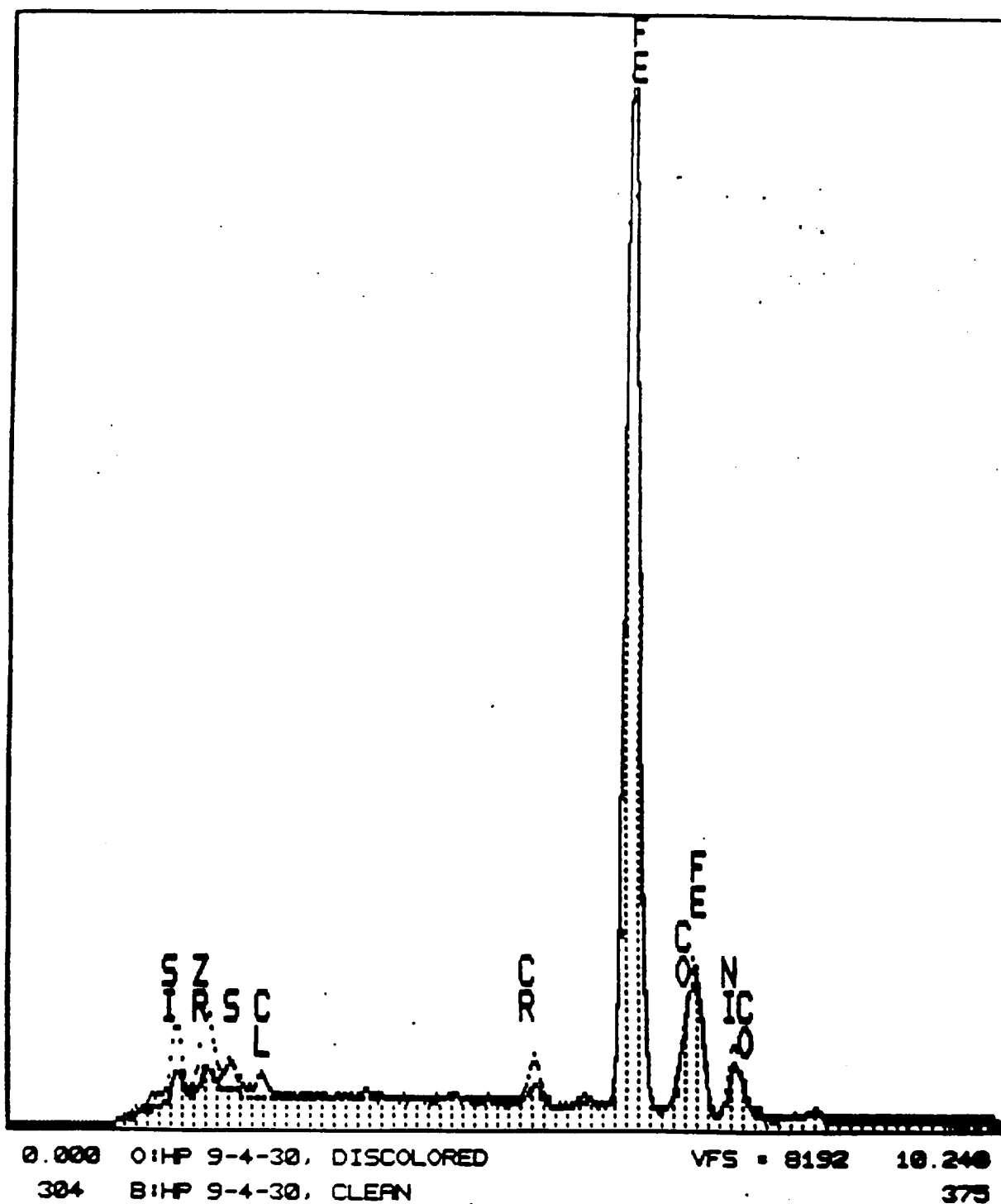


FIGURE XII
SEM/EDAX ANALYSIS
CLEAN* VS. DISCOLORED HP9-4-30 STEEL**

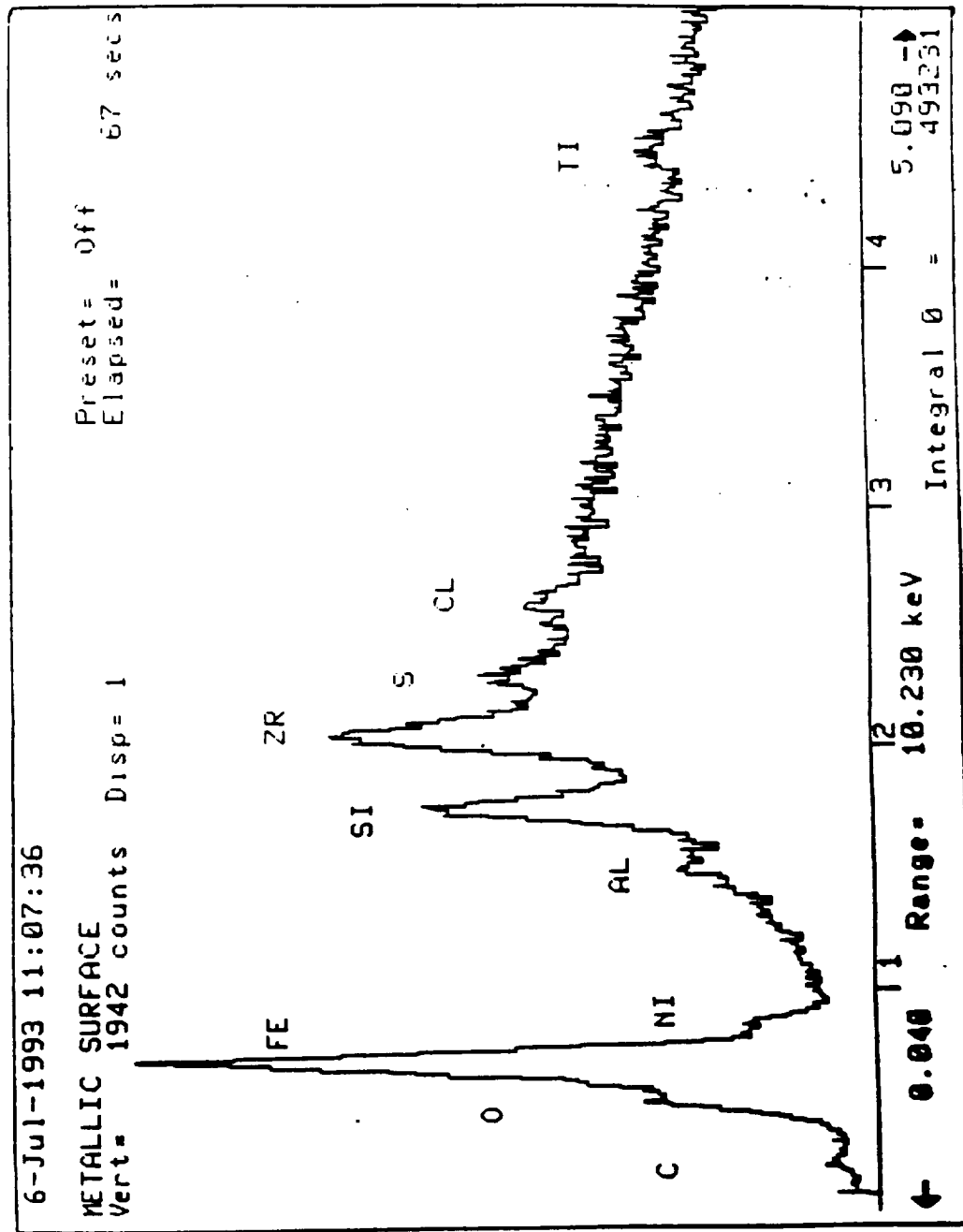
(ANALYSIS PERFORMED BY JAMES COSTON, EH22)



*PANELS CLEANED WITH TURCO 3878 LF-NC AND DID NOT DISCOLOR.

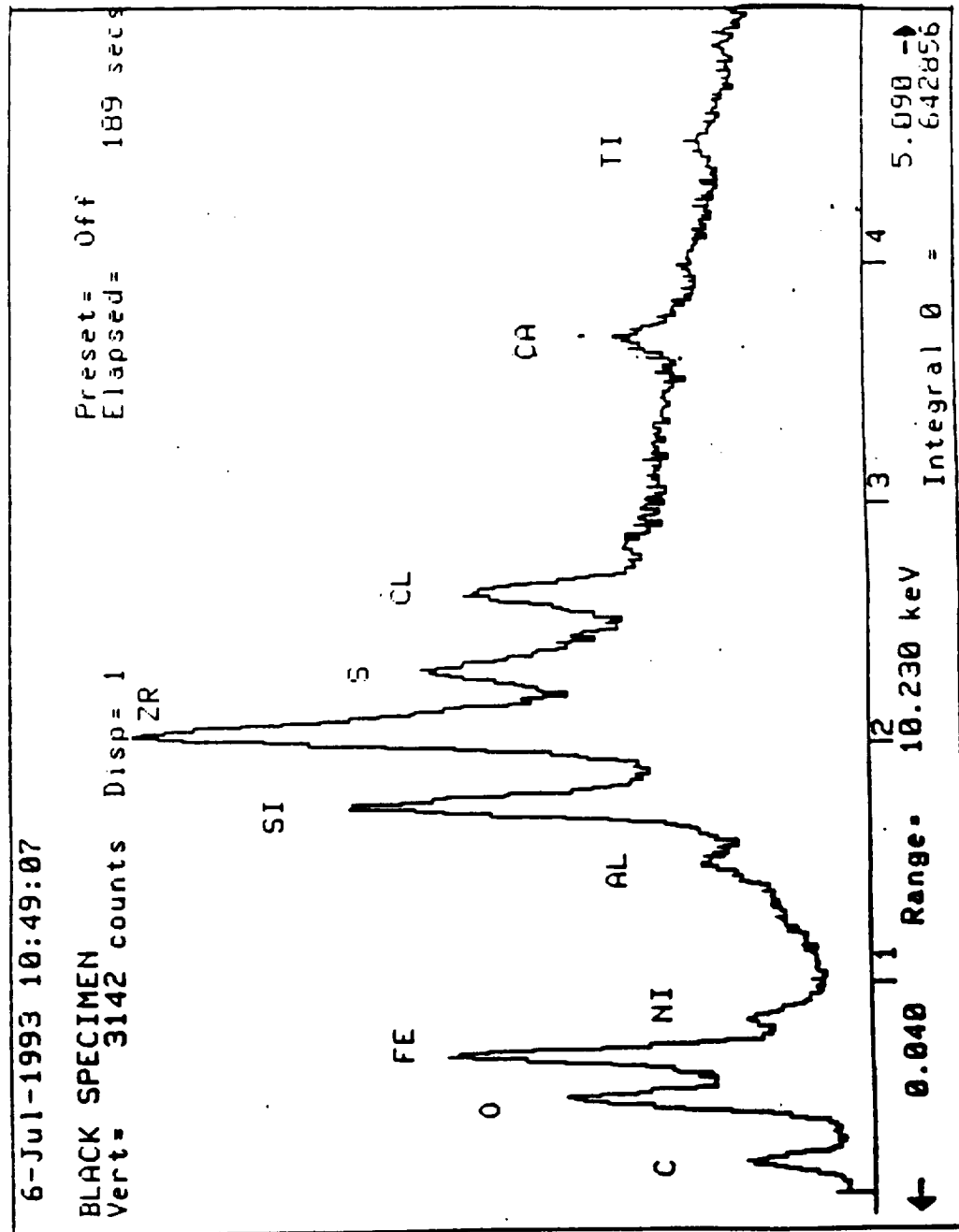
**PANELS DISCOLORED DURING CLEANING WITH TURCO 3878 LF-NC.

FIGURE XIII
SEM/EDAX ANALYSIS OF CLEAN HP9-4-30 STEEL



PANELS CLEANED WITH TURCO 3878 LF-NC AND DID NOT DISCOLOR.
 ANALYSIS PERFORMED BY JEFF SANDERS, ITRI, MSFC

FIGURE XIV
SEM/EDX ANALYSIS OF DISCOLORED HP9-4-30
STEEL



PANELS DISCOLORED DURING CLEANING WITH TURCO 3878 LF-NC.
 ANALYSIS PERFORMED BY JEFF SANDERS, ITRI, MSFC

FIGURE XV: HP9-4-30 RUN 2, 70F/40%RH/48HR + 75F/55%RH/24HR

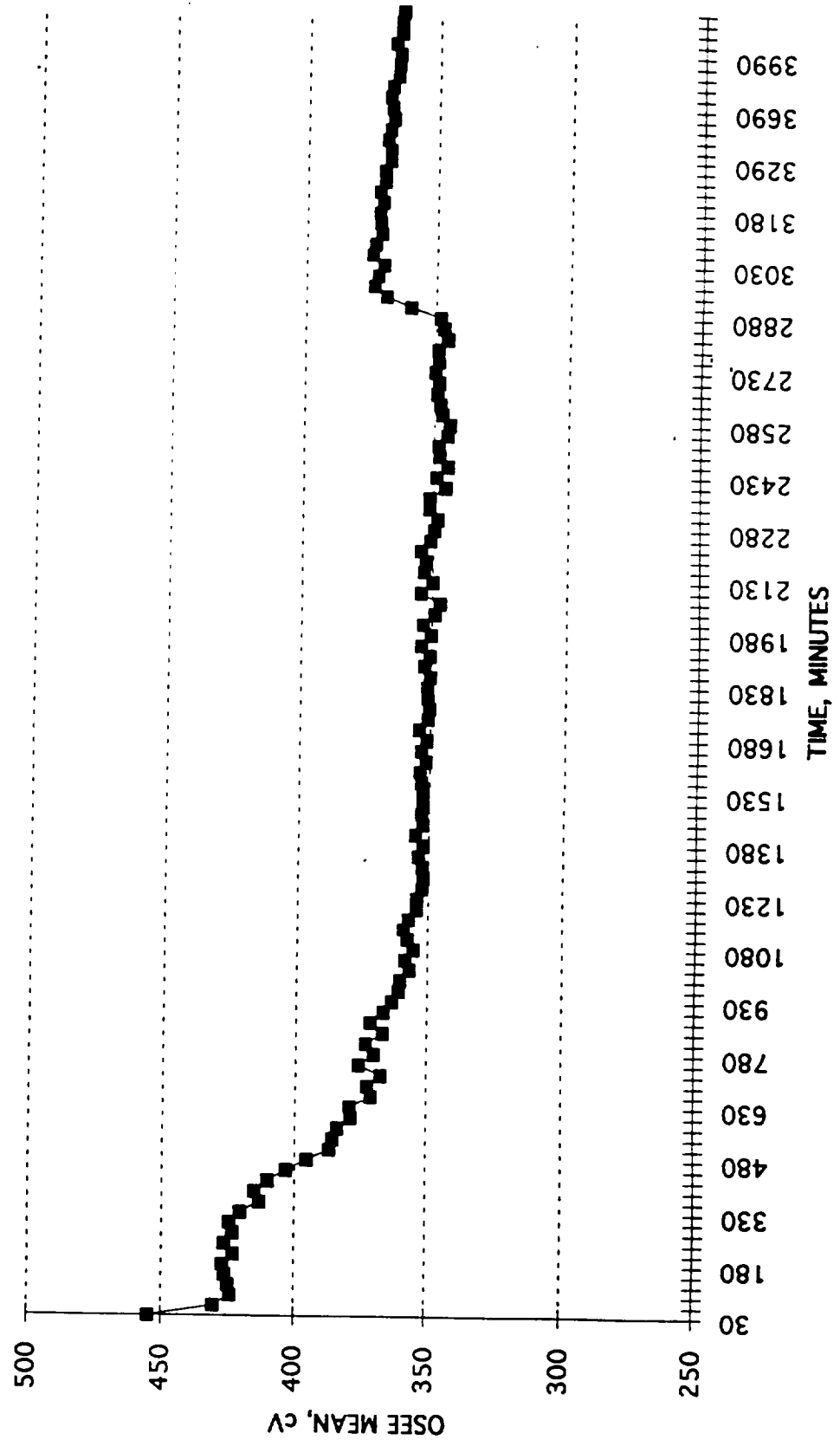


FIGURE XVI: HP9-4-30 RUN 4, 70F/75%RH/48HRS + 75F/55%RH/24HRS

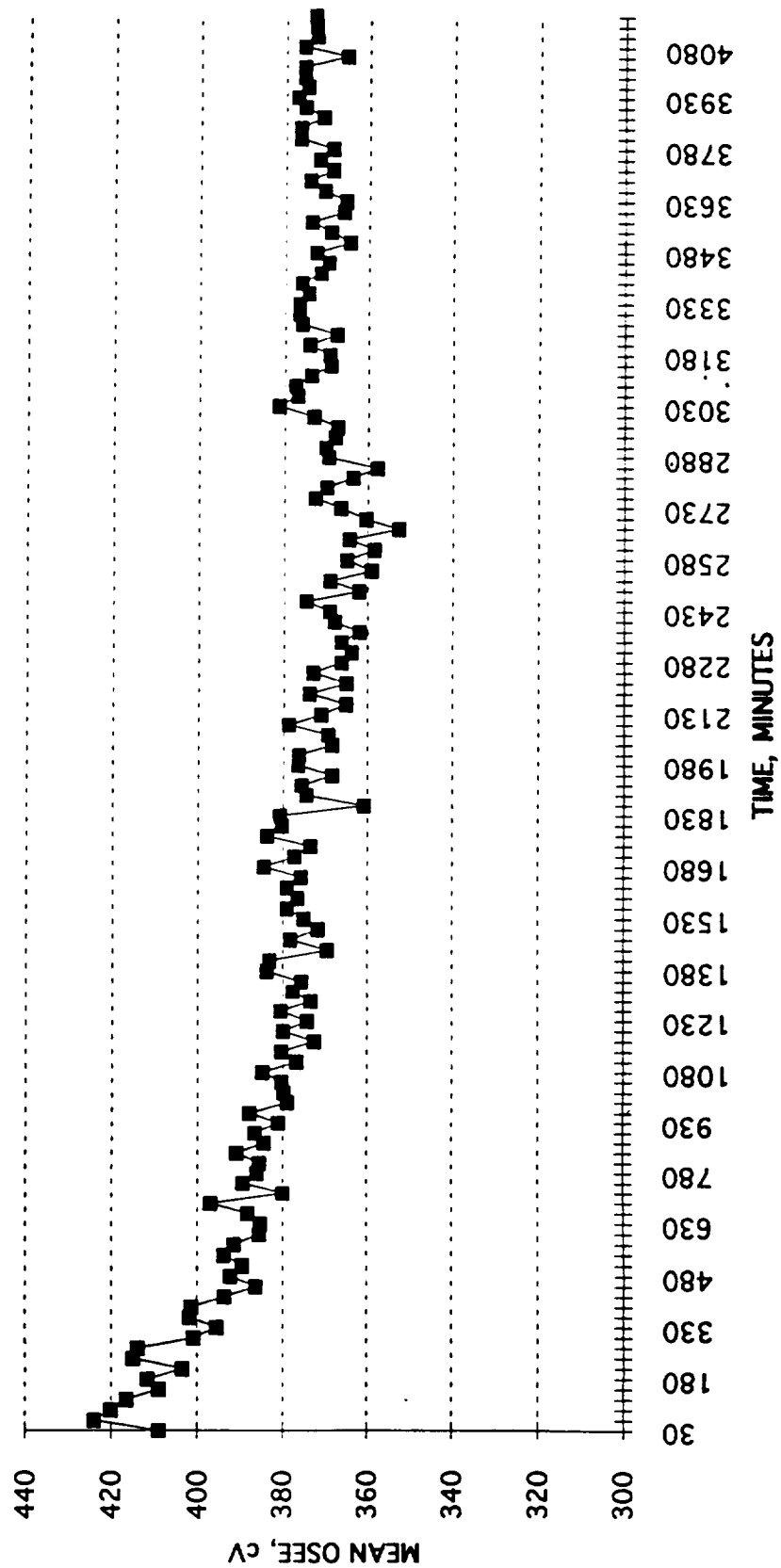


TABLE I
EFFECT OF AGING ON D6AC/NBR BOND
STRENGTH AFTER EXTREME ENVIRONMENTAL
EXPOSURE

<u>PANEL NUMBER</u>	<u>PRE-BONDING CONDITIONING</u>	<u>POST- BONDING AGING</u>	<u>HARDNESS SHORE A</u>	<u>AVERAGE PEEL STRENGTH PLI*</u>	<u>AVERAGE TENSILE STRENGTH PSI*</u>
E1	50F/20RH/3HRS	-	59-75	218	713
E2	50F/20RH/7DAYS	-	65-73	218	759
E3	50F/60RH/3HRS	-	68-75	217	717
E4	50F/60RH/7DAYS	-	67-73	221	742
E5	100F/20RH/3HRS	-	65-75	221	692
E6	100F/20RH/7DAYS	-	63-73	212	668
E7	100F/60RH/3HRS	-	65-75	217	718
E8	100F/60RH/7DAYS	-	67-73	224	802
T001	FRESHLEY CLEANED D6AC	-	65-73	209	675
T002	FRESHLEY CLEANED D6AC	-	68-73	207	673
T003	FRESHLEY CLEANED D6AC	-	66-72	204	682
T004	FRESHLEY D6AC	-	63-72	209	621
T005	FRESHLEY D6AC	-	62-73	201	711
T006	FRESHLEY CLEANED D6AC	-	65-74	197	718
E83MB	100F/60RH/7DAYS	3 MO. AMBIENT TEMP. AND RH	69-75/68-75**	216	707
E83MA	100F/60RH/7DAYS	6 MO. AMBIENT TEMP. AND RH	67-71/65-72**	215	677
E86MB	100F/60RH/7DAYS	12 MO. AMBIENT TEMP. AND RH	66-71/65-73**	222	719

* All specimens exhibited 100% cohesive insulation failures. Peel adhesion tested at 90° and at 2 inches per minute. Tensile adhesion tested at 0.5 inches per minute.

** Before aging hardness/After aging hardness

TABLE II
TAGUCHI DESIGN FOR 2219-T87 ALUMINUM ENVIRONMENTAL TESTS

<u>FACTORS</u>		
<u>A</u>	<u>B</u>	<u>C</u>
TEMP	REL. HUMIDITY	EXPOSURE TIME
LEVELS		
$T_1 = \underline{65^\circ\text{F}}$	$\text{RH}_1 = \underline{20\%}$	$t_1 = \underline{48 \text{ Hrs}}$
$T_2 = \underline{100^\circ\text{F}}$	$\text{RH}_2 = \underline{60\%}$	$t_2 = \underline{96 \text{ Hrs}}$

TAGUCHI L8 ORTHOGONAL ARRAY (RESOLUTION 4, ALL MAIN EFFECTS / INTERACTIONS CAN BE ESTIMATED)

RUN	<u>FACTORS</u>							<u>RESPONSES</u>		
	1 A	2 B	3 AxB	4 C	5 AxC	6 BxC	7 AxBxC	OSEE	OSEE Δ	TA Av.3
1	1	1	1	1	1	1	1	O ₁	D ₁	T ₁
2	1	1	1	2	2	2	2	O ₂	D ₂	T ₂
3	1	2	2	1	1	2	2	O ₃	D ₃	T ₃
4	1	2	2	2	2	1	1	O ₄	D ₄	T ₄
5	2	1	2	1	2	1	2	O ₅	D ₅	T ₅
6	2	1	2	2	1	2	1	O ₆	D ₆	T ₆
7	2	2	1	1	2	2	1	O ₇	D ₇	T ₇
8	2	2	1	2	1	1	2	O ₈	D ₈	T ₈

*T.A.-Tape adhesion test

TABLE III
APPROXIMATE ABSOLUTE MOISTURES AND DEW POINTS
FOR
2219-T87 ALUMINUM ENVIRONMENTAL EXPOSURE CONDITIONS

<u>T °F</u>	<u>RH %</u>	<u>ABSOLUTE MOISTURE</u> <u>PPM</u>	<u>GRAINS</u>	<u>D. P. °F</u>
100	60	~40,000	~176	~85
100	20	~15,000	~59	~58
65	60	~12000	~57	~55
65	20	~3800	~19	~25

TAGUCHI OUTER ARRAY DESIGN FOR 2219-T87 ALUMINUM CONTAMINATION TESTS

Contaminant: A= Kaydol Oil
B= CRC 2-26 Oil

Concentration (mg/ft2):	1	2	3
	1	5	10

***T.A.-Tape adhesion test**

TABLE V **EFFECT OF ENVIRONMENTAL EXPOSURE/CONTAMINATION** **ON 2219-T87 ALUMINUM OSEE RESPONSE AND PRIMER** **ADHESION**

TEST SPECIMEN FLOW: Taguchi Run____/____°F/____%RH

- Note:** 1. Approved gloves to be used in the handling of panels.
 2. A log will be kept for each set of panels in which all pertinent data shall be recorded.

Date

- ____ 1. *Verify Test & Witness Panel Configurations & Serial Numbers*
 -Size:
 1 ea. Test Panel - 8"x12"x1/8"
 3 ea. Witness Panels - 3"x3"x1/8"
 -Serial Numbers:
 Environmental Test Panel____; Witness Coupons____,____,____
 Contamination Test Panels:____; Witness Coupons____,____,____
 ____; Witness Coupons____,____,____
 ____; Witness Coupons____,____,____
 ____; Witness Coupons____,____,____
 ____; Witness Coupons____,____,____
- ____ 2. *Clean and Deoxidize Panels*
 -Hand wipe with MEK
 -Vapor degrease 2 minutes with perchloroethylene
 Time In:____
 Time Out:____
 -Alkaline clean with Turco 4215 (140-170°F) for 15 minutes
 Time In:____
 Time Out:____
 -Hot water rinse for 2 minutes
 Start rinse:____
 Rinse completed:____
 -Deoxidize with Smutgo #1 for 20 minutes
 Time In:____
 Time Out:____
 -Rinse with cold DI water for 5 minutes
 Start rinse:____
 Rinse completed:____
 -Oven dry at 140°F for 5 minutes
 Time In:____
 Time Out:____

- ____ 3. *Prepare Environmental Chamber & OSEE System (w/ controlled z-axis scanning).* NOTE: OSEE values not corrected for grains of moisture
- Run OSEE System: Check w/ Calibration surface
 - Calibration Std.: Type _____ ID# _____
 - OSEE Value: mean _____ std.dev. _____
 - Set Controls for Chamber
 - Temperature _____ °F/R.H. _____ %/Exposure Duration _____
 - Record OSEE, Chamber Settings & Barometric Pressure in Log Book
 - Scan of Calibration Std. in chamber
 - Type _____ ID# _____; mean _____ std.dev. _____
 - Initial scan of panel after cleaning (Table I):
- | | | | | |
|-------------|------------|------------|----------------|-------------------------|
| Panel _____ | Time _____ | mean _____ | std.dev. _____ | Placed in Chamber _____ |
| Panel _____ | Time _____ | mean _____ | std.dev. _____ | Placed in Chamber _____ |
| Panel _____ | Time _____ | mean _____ | std.dev. _____ | Placed in Chamber _____ |
| Panel _____ | Time _____ | mean _____ | std.dev. _____ | Placed in Chamber _____ |
| Panel _____ | Time _____ | mean _____ | std.dev. _____ | Placed in Chamber _____ |
| Panel _____ | Time _____ | mean _____ | std.dev. _____ | Placed in Chamber _____ |
| Panel _____ | Time _____ | mean _____ | std.dev. _____ | Placed in Chamber _____ |

- ____ 4. *Initiate Environmental Exposure*
- Close Chamber Door and Log Time When Chamber Reaches Specified Conditions: TEST START TIME _____

- ____ 5. *OSEE Measurements*
- Make 1st Scan (Panel _____) When Chamber Reaches Test Conditions
 - Verify Time (Log if different from time chamber reached test conditions)
 - Set System to Repeat Scan Every _____ minutes for _____ hrs, then every _____ hours until end of test.

- ____ 6. *Remove Witness Coupons*
- Witness Coupons removed, Bagged in GN2 and distributed for analysis as follows:

<u>Serial Numbers</u>						<u>Time</u>	<u>Recipient</u>
____	____	____	____	____	____	____	____
____	____	____	____	____	____	____	____
____	____	____	____	____	____	____	____

- Denote SN and Time Removed on Bag and in Log Book

- ____7. *Termination of Environmental Exposure*
-Remove Test Panel from Chamber and Log Time____
-OSEE scan (Table I):
Panel____Time____mean____std.dev.____
Panel____Time____mean____std.dev.____
Panel____Time____mean____std.dev.____
Panel____Time____mean____std.dev.____
Panel____Time____mean____std.dev.____
Panel____Time____mean____std.dev.____
Panel____Time____mean____std.dev.____

- ____8. *Contaminate panel (if applicable):*
- **Panel**____
-Contaminant type____
-Desired concentration____mg/ft²
 -Prepare witness aluminum foil
 -Weight of foil____
 -Spray uniform coat of contaminant over test panel and witness foil
 -Allow ____minutes for carrier solvent to evaporate
 -Weight of foil after contaminant applied____
 -Total contaminant wt.____ Concentration in mg/ft²____
-OSEE scan after contamination: TIME/TEMP/RH____/____/____
 mean=____ std.dev.=____

- **Panel**____
-Contaminant type____
-Desired concentration____mg/ft²
 -Prepare witness aluminum foil
 -Weight of foil____
 -Spray uniform coat of contaminant over test panel and witness foil
 -Allow ____minutes for carrier solvent to evaporate
 -Weight of foil after contaminant applied____
 -Total contaminant wt.____ Concentration in mg/ft²____
-OSEE scan after contamination: TIME/TEMP/RH____/____/____
 mean=____ std.dev.=____

- **Panel** _____
-Contaminant type _____
-Desired concentration _____ mg/ft²
 -Prepare witness aluminum foil
 -Weight of foil _____
 -Spray uniform coat of contaminant over test panel and witness foil
 -Allow _____ minutes for carrier solvent to evaporate
 -Weight of foil after contaminant applied _____
 -Total contaminant wt. _____ Concentration in mg/ft² _____
-OSEE scan after contamination: TIME/TEMP/RH _____/_____/_____
 mean=_____ std.dev.=_____

- **Panel** _____
-Contaminant type _____
-Desired concentration _____ mg/ft²
 -Prepare witness aluminum foil
 -Weight of foil _____
 -Spray uniform coat of contaminant over test panel and witness foil
 -Allow _____ minutes for carrier solvent to evaporate
 -Weight of foil after contaminant applied _____
 -Total contaminant wt. _____ Concentration in mg/ft² _____
-OSEE scan after contamination: TIME/TEMP/RH _____/_____/_____
 mean=_____ std.dev.=_____

- **Panel** _____
-Contaminant type _____
-Desired concentration _____ mg/ft²
 -Prepare witness aluminum foil
 -Weight of foil _____
 -Spray uniform coat of contaminant over test panel and witness foil
 -Allow _____ minutes for carrier solvent to evaporate
 -Weight of foil after contaminant applied _____
 -Total contaminant wt. _____ Concentration in mg/ft² _____
-OSEE scan after contamination: TIME/TEMP/RH _____/_____/_____
 mean=_____ std.dev.=_____

- **Panel**_____
- Contaminant type_____
- Desired concentration_____mg/ft²
 - Prepare witness aluminum foil
 - Weight of foil_____
 - Spray uniform coat of contaminant over test panel and witness foil
 - Allow _____minutes for carrier solvent to evaporate
 - Weight of foil after contaminant applied_____
 - Total contaminant wt._____ Concentration in mg/ft²_____
- OSEE scan after contamination: TIME/TEMP/RH_____/_____/_____
 - mean=_____ std.dev.=_____

- _____9. *Mix , Apply and Cure Primer According to Martin-Marietta PI-3003 and 3003-1*
- Verify that mixing area is within PI limits of 65-100° temperature and ≤ 70% RH. Document primer expiration date and mix date.
 - Temp_____/RH_____
 - Primer Expiration Date_____/Mix Date_____
 - Prior to opening, shake the base component in the container in which it was received using a paint shaker.
 - Examine base and curing component for grit, seediness, skins, lumps, abnormal thickness or livering which can not be readily mixed to form a smooth mixture.
 - Strain each primer component prior to or during mixing through paint strainer MMC02100375 or MMC02205500.
 - Measure 4 volumes of base component into a clean container or a pressure pot. Stirring constantly, slowly add 1 volume of curing solution, then slowly add 4 volumes of solvent reducer. Stir until homogeneous.
 - Vol. Base Component_____ Vol. Solvent Reducer_____
 - Vol. Curing Solution_____
 - Time Mix Completed (all components added)_____
 - Using a #1 Zahn cup and stop watch, check primer viscosity within 1 hour after mixing. Viscosity shall be 28 to 42 seconds at 70 to 100°F. Add additional solvent reducer, if required, to bring viscosity into required range, mixing until homogeneous. Solvent reducer addition shall not exceed 10% of total primer mix. Solvent Reducer added for viscosity adjustment can only be added at time of original viscosity check.
 - Primer Viscosity/Time of Measurement_____/_____
 - Added Solvent reducer volume/Time_____/_____
 - Cover container with tight fitting lid when not in use. Allow mixed primer to stand at least 50 minutes prior to application.

-Freshly mixed primer may be stored under refrigeration for a maximum of 36 hours provided the requirements specified in PI-3003 Basic are met.

Mix Stored: Yes____/No____

Storage Conditions: Time into Storage____

Storage Temperature____

Time Out of Storage____

-Document temperature, RH conditions of spray area, application date and time: Temperature/RH____/____;Date/Time____/____

-Pressurize spray system to the required pressure, 30 psi max., and adjust spray pattern as required.

-Spray apply primer to panel at a thickness of no greater than 0.0025 in.

-Cure primer by either of the following procedures:

-65°F minimum and 70%RH maximum for 16 hours

-65°F minimum and 70%RH maximum for 1 hour to flash off volatile solvents, followed by either 4 hr. minimum at 115-130°F or 2 hr. minimum at 130-150°F.

Start/Finish Ambient Cure____/____

Start/Finish solvent flash time at 65°F____/____

Start/Finish cure at 115-130°F____/____

Start/Finish cure at 130-150°F____/____

____ 10. *Perform Wet Tape Test According to PI-3003-1(Wet Pad Method)*

-Place water soaked pad made from nymple cloth, not less than 3"x3", against primed surface.

-Cover pad with polyethylene and seal edges with tape MMSJ414AXXX or MMSJ562XXX. Tape type____

-After 2 hours minimum, remove wet patch and immediately dry surface with clean, dry wiping cloth, MMS4968AXXX.

-Within 3 minutes, test the primed area where the pad was removed by applying strips of tape, MMSJ431A100, 3" to 8" in length to the surface, pressing tape down with firm hand pressure, then removing the tape in one abrupt motion.

-Examine area for primer damage. Removal of more than 2% of the primer in test area shall be considered unacceptable.

-If specimen fails, rework as specified in PI.

-Document results in log book

Panel____Time patch applied____/Removed____:Pass____or Fail____

Panel____Time patch applied____/Removed____:Pass____or Fail____

Panel____Time patch applied____/Removed____:Pass____or Fail____

Panel____Time patch applied____/Removed____:Pass____or Fail____

Panel____Time patch applied____/Removed____:Pass____or Fail____

Panel____Time patch applied____/Removed____:Pass____or Fail____

Panel____Time patch applied____/Removed____:Pass____or Fail____

TABLE VI
CONTRACT NAS8-39244
TAGUCHI DESIGN FOR 2219-T87 ALUMINUM ENVIRONMENTAL TESTS

FACTORS

	<u>A</u>	<u>B</u>	<u>C</u>
	TEMP	REL. HUMIDITY	EXPOSURE TIME
LEVELS	$T_1 = \underline{65^\circ\text{F}}$	$RH_1 = \underline{20\%}$	$t_1 = \underline{48 \text{ Hrs}}$
	$T_2 = \underline{100^\circ\text{F}}$	$RH_2 = \underline{60\%}$	$t_2 = \underline{96 \text{ Hrs}}$

TAGUCHI L8 ORTHOGONAL ARRAY (RESOLUTION 4, ALL MAIN EFFECTS / INTERACTIONS CAN BE ESTIMATED)

RUN	FACTORS								RESPONSES	
	1 A	2 B	3 AxB	4 C	5 AxC	6 BxC	7 AxBxC	ENV. CHAMBER QSEEA	TA AV.3	
1	1	1	1	1	1	1	1	72	PASS	
2	1	1	1	2	2	2	2	230	PASS	
3	1	2	2	1	1	2	2	120	PASS	
4	1	2	2	2	2	1	1	180	PASS	
5	2	1	2	1	2	1	2	570	PASS	
6	2	1	2	2	1	2	1	560	PASS	
7	2	2	1	1	2	2	1	590	PASS	
8	2	2	1	2	1	1	2	830	PASS	

*T.A.-Tape adhesion test

TABLE VII
2219-T87 ALUMINUM ENVIRONMENTAL
EXPOSURE/CONTAMINATION STUDY

TAGUCHI RUN 1: 65°F/20%RH/48HRS

<u>PANEL</u>	<u>INITIAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>POST-</u> <u>EXPOSURE</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>Δ</u> <u>OSEE</u> <u>(cV)</u>	<u>CONTAMINANT</u> <u>TYPE/</u> <u>QUANTITY</u> <u>(MG/FT²)</u>	<u>FINAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV.</u> <u>(cV)</u>	<u>WET TAPE</u> <u>PRIMER</u> <u>ADHESION</u> <u>TEST</u>
EA1	1168/33	1044/15	124	N/A	N/A	PASS
EACA11	1201/18	1079/22	122	KAYDOL/0.9	1004/25	PASS
EACA21	1230/24	1063/13	167	KAYDOL/4.5	679/67	PASS
EACA31	1194/30	1035/26	159	KAYDOL/9.7	195/89	PASS
EACB11	1223/21	1065/15	158	CRC Si/1.0	744/68	PASS
EACB21	1224/22	1068/10	156	CRC Si/5.2	229/32	PASS
EACB31	1218/21	1066/13	152	CRC Si/9.7	54/25	PASS
Mean	1208/24	1060/16	148			

TAGUCHI RUN 2: 65°F/20%RH/96HRS

<u>PANEL</u>	<u>INITIAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>POST-</u> <u>EXPOSURE</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>Δ</u> <u>OSEE</u> <u>(cV)</u>	<u>CONTAMINANT</u> <u>TYPE/</u> <u>QUANTITY</u> <u>(MG/FT²)</u>	<u>FINAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV.</u> <u>(cV)</u>	<u>WET TAPE</u> <u>PRIMER</u> <u>ADHESION</u> <u>TEST</u>
EA2	1090/45	851/77	239	N/A	N/A	PASS
EACA12	957/54	667/58	290	KAYDOL/1.0	467/98	PASS
EACA22	1066/77	819/76	247	KAYDOL/4.6	252/69	PASS
EACA32	1099/36	868/58	231	KAYDOL/10.2	169/50	FAIL
EACB12	1048/60	792/79	256	CRC Si/1.2	296/126	PASS
EACB22	980/63	704/80	276	CRC Si/5.4	115/32	FAIL
EACB32	1098/42	833/52	265	CRC Si/10.0	48/26	PASS
Mean	1048/54	791/69	258			

TAGUCHI RUN 3: 65°F/60%RH/48HRS

<u>PANEL</u>	<u>INITIAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>POST-</u> <u>EXPOSURE</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>Δ</u> <u>OSEE</u> <u>(cV)</u>	<u>CONTAMINANT</u> <u>TYPE/</u> <u>QUANTITY</u> <u>(MG/FT²)</u>	<u>FINAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV.</u> <u>(cV)</u>	<u>WET TAPE</u> <u>PRIMER</u> <u>ADHESION</u> <u>TEST</u>
EA3	1060/25	974/32	86	N/A	N/A	PASS
EACA13	1075/27	961/28	114	KAYDOL/1.3	868/54	PASS
EACA23	1079/24	1011/43	68	KAYDOL/5.2	523/132	FAIL
EACA33	1087/24	1019/28	68	KAYDOL/10.6	260/118	PASS
EACB13	1074/22	993/42	81	CRC Si/1.3	623/107	PASS
EACB23	1072/17	983/21	89	CRC Si/5.7	88/31	PASS
EACB33	1073/25	978/38	95	CRC Si/9.7	28/19	FAIL
Mean	1074/23	988/33	100			

TAGUCHI RUN 4 : 65°F/60%RH/96HRS

<u>PANEL</u>	<u>INITIAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>POST-</u> <u>EXPOSURE</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>Δ</u> <u>OSEE</u> <u>(cV)</u>	<u>CONTAMINANT</u> <u>TYPE/</u> <u>QUANTITY</u> <u>(MG/FT²)</u>	<u>FINAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV.</u> <u>(cV)</u>	<u>WET TAPE</u> <u>PRIMER</u> <u>ADHESION</u> <u>TEST</u>
EA4	1010/47	872/70	138	N/A	N/A	PASS
EACA14	967/41	694/69	273	KAYDOL/0.9	638/65	PASS
EACA24	985/68	840/89	145	KAYDOL/4.8	551/95	PASS
EACA34	941/43	902/36	39	KAYDOL/10.0	340/86	PASS
EACB14	962/44	778/52	184	CRC Si/1.2	437/68	PASS
EACB24	933/69	647/91	286	CRC Si/4.9	195/39	PASS
EACB34	953/41	719/44	234	CRC Si/10.0	68/30	PASS
Mean	964/50	779/64	186			

TAGUCHI RUN 5: 100°F/20%RH/48HRS

<u>PANEL</u>	<u>INITIAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>POST-</u> <u>EXPOSURE</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>Δ</u> <u>OSEE</u> <u>(cV)</u>	<u>CONTAMINANT</u> <u>TYPE/</u> <u>QUANTITY</u> <u>(MG/FT²)</u>	<u>FINAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV.</u> <u>(cV)</u>	<u>WET TAPE</u> <u>PRIMER</u> <u>ADHESION</u> <u>TEST</u>
EA5	1206/41	932/32	274	N/A	N/A	PASS
EACA15	1184/34	883/50	301	KAYDOL/0.9	846/53	PASS
EACA25	1115/46	821/46	294	KAYDOL/5.2	581/73	PASS
EACA35	1166/43	900/42	266	KAYDOL/9.6	290/135	PASS
EACB15	1190/44	966/55	224	CRC Si/0.9	771/89	PASS
EACB25	1188/41	923/51	265	CRC Si/5.5	250/79	PASS
EACB35	1145/69	1016/38	129	CRC Si/9.6	85/46	FAIL
Mean	1171/45	920/45	250			

TAGUCHI RUN 6: 100°F/20%RH/96HRS

<u>PANEL</u>	<u>INITIAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>POST-</u> <u>EXPOSURE</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>Δ</u> <u>OSEE</u> <u>(cV)</u>	<u>CONTAMINANT</u> <u>TYPE/</u> <u>QUANTITY</u> <u>(MG/FT²)</u>	<u>FINAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV.</u> <u>(cV)</u>	<u>WET TAPE</u> <u>PRIMER</u> <u>ADHESION</u> <u>TEST</u>
EA6	1074/48	986/69	88	N/A	N/A	PASS
EACA16	1103/20	1061/39	42	KAYDOL/1.05	990/44	PASS
EACA26	1097/21	1119/34	22(1)	KAYDOL/4.6	614/154	PASS
EACA36	1100/20	1124/30	24(1)	KAYDOL/9.3	224/90	PASS
EACB16	1090/25	993/53	97	CRC Si/1.05	801/74	PASS
EACB26	1008/98	934/113	74	CRC Si/5.2	271/91	PASS
EACB36	1082/44	999/108	83	CRC Si/9.9	97/68	PASS
Mean	1079/39	1031/64	49			

TAGUCHI RUN 6: 100°F/20%RH/96HRS (REPEAT)

<u>PANEL</u>	<u>INITIAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>POST-</u> <u>EXPOSURE</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>Δ</u> <u>OSEE</u> <u>(cV)</u>	<u>CONTAMINANT</u> <u>TYPE/</u> <u>QUANTITY</u> <u>(MG/FT²)</u>	<u>FINAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV.</u> <u>(cV)</u>	<u>WET TAPE</u> <u>PRIMER</u> <u>ADHESION</u> <u>TEST</u>
REA6	1144/19	1092/37	52	N/A	N/A	PASS
RA16	1165/21	1053/38	112	KAYDOL/1.2	756/63	PASS
RA26	1136/24	984/45	152	KAYDOL/4.5	397/80	PASS
RA36	1133/17	1084/52	49	KAYDOL/9.6	134/45	PASS
RB16	1141/17	1063/26	78	CRC Si/1.3	425/129	PASS
RB26	1137/12	1004/22	133	CRC Si/6.2	107/38	PASS
RB36	1137/13	1011/25	126	CRC Si/13.0	31/12	FAIL
Mean	1142/18	1042/35	100			

TAGUCHI RUN 7: 100°F/60%RH/48HRS

<u>PANEL</u>	<u>INITIAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>POST-</u> <u>EXPOSURE</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>Δ</u> <u>OSEE</u> <u>(cV)</u>	<u>CONTAMINANT</u> <u>TYPE/</u> <u>QUANTITY</u> <u>(MG/FT²)</u>	<u>FINAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV.</u> <u>(cV)</u>	<u>WET TAPE</u> <u>PRIMER</u> <u>ADHESION</u> <u>TEST</u>
EA7	1105/26	831/40	274	N/A	N/A	PASS
EACA17	1053/23	886/33	167	KAYDOL/1.3	766/38	PASS
EACA27	1081/32	861/62	220	KAYDOL/4.9	452/101	PASS
EACA37	1157/18	956/57	201	KAYDOL/9.6	213/89	PASS
EACB17	1094/36	931/43	163	CRC Si/1.2	614/66	PASS
EACB27	1092/29	945/43	147	CRC Si/4.5	239/63	FAIL
EACB37	1098/62	940/93	158	CRC Si/9.5	70/42	PASS
Mean	1097/32	907/53	190			

TAGUCHI RUN 8: 100°F/60%RH/96HRS

<u>PANEL</u>	<u>INITIAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>POST-</u> <u>EXPOSURE</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV.</u> <u>(cV)</u>	<u>Δ</u> <u>OSEE</u> <u>(cV)</u>	<u>CONTAMINANT</u> <u>TYPE/</u> <u>QUANTITY</u> <u>(MG/FT²)</u>	<u>FINAL</u> <u>OSEE:</u> <u>MEAN/</u> <u>STD.DEV</u> <u>(cV)</u>	<u>WET TAPE</u> <u>PRIMER</u> <u>ADHESION</u> <u>TEST</u>
EA8	1053/48	977/53	76	N/A	N/A	PASS
EACA18	1064/22	961/34	103	KAYDOL/1.05	863/56	PASS
EACA28	1054/28	951/66	103	KAYDOL/6.0	480/121	PASS
EACA38	1053/20	997/31	56	KAYDOL/9.9	115/66	PASS
EACB18	1063/33	918/64	145	CRC Si/1.05	763/53	PASS
EACB28	1042/37	909/55	133	CRC Si/4.6	339/110	FAIL
EACB38	1038/43	885/55	153	CRC Si/9.75	53/53	FAIL
Mean	1052/33	943/51	110			

NOTE: ALL OSEE MEASUREMENTS TAKEN USING THE LAB ENVIRONMENT
(GENERATION II) OSEE SYSTEM.

AC9g/3/93

TABLE VIII
ENVIRONMENTAL EXPOSURE RESULTS SUMMARY

Panel Set	Exposure Conditions	Table 1 Results****			Env. Chamber Results		
		OSEE Before Exposure	OSEE After Exposure	Delta OSEE	Zero Time*** OSEE	Final OSEE	Delta OSEE
EA1	65F/20%RH/48H	1168	1044	124	472	400	72
REA1*	65F/20%RH/48H	1140	967	173	2267	1538	729
EA2	65F/20%RH/96HR	1090	851	239	570	340	230
EA3**	65F/60%RH/48H	1060	974	86	1938	1818	120
EA4	65F/60%RH/96HR	1010	872	138	355	175	180
EA5	100F/20%RH/48HR	1206	932	274	1270	700	570
EA6	100F/20%RH/96HR	1074	986	88	1540	920	620
REA6	100F/20%RH/96HR	1144	1092	52	1690	1130	560
EA7	100F/60%RH/48HR	1105	831	274	1170	580	590
EA8	100F/60%RH/96HR	1053	977	76	1640	810	830

* New UV bulb and power supply installed prior to this Run.

** New UV bulb installed in chamber prior to this Run.

*** Zero time measurement taken after environmental chamber had reached target exposure conditions; typically 15-20 minutes after panel inserted.

**** All OSEE responses are in cV.

AC9h/3/93

TABLE IX
WET TAPE TEST RESULTS VS. COATING LEVEL/TYPE, 2219-T87
ALUMINUM

<u>PANEL SET</u>	<u>BASE-LINE</u>	<u>1 MG/FT² KAYDOL</u>	<u>5 MG/FT² KAYDOL</u>	<u>10 MG/FT² KAYDOL</u>	<u>1 MG/FT² SILICONE</u>	<u>5 MG/FT² SILICONE</u>	<u>10 MG/FT² SILICONE</u>
EA1	PASS	PASS	PASS	PASS	PASS	PASS	PASS
EA2	PASS	PASS	PASS	FAIL	PASS	FAIL	PASS
EA3	PASS	PASS	FAIL	PASS	PASS	PASS	FAIL
EA4	PASS	PASS	PASS	PASS	PASS	PASS	PASS
EA5	PASS	PASS	PASS	PASS	PASS	PASS	FAIL
EA6	PASS	PASS	PASS	PASS	PASS	PASS	PASS
EA7	PASS	PASS	PASS	PASS	PASS	PASS	FAIL
EA8	PASS	PASS	PASS	PASS	PASS	FAIL	FAIL

WET TAPE TEST PERFORMED PER MARTIN MARIETTA PROCEDURE MPI PI-3003-1

AC93TIMI

TABLE X
WATER BREAK FREE TESTING OF 2219-T87 ALUMINUM COATED
WITH KAYDOL OR SILICONE

<u>COATING</u>	<u>LEVEL, MG/FT²</u>	<u>INITIAL OSEE, CV</u>	<u>FINAL OSEE, CV</u>	<u>DELTA OSEE, CV</u>	<u>WATER BREAK</u>
KAYDOL	1.2	1019	959	60	PASS
KAYDOL	1.95	1000	788	212	FAIL
KAYDOL	4.35	912	474	438	FAIL
KAYDOL	5.7	1022	552	470	FAIL
KAYDOL	9.75	967	105	862	FAIL
KAYDOL	12.0	904	32	872	FAIL
SILICONE	0.9	1076	945	131	PASS
SILICONE	2.6	1192	623	569	PASS
SILICONE	4.2	948	335	613	PASS
SILICONE	4.8	1022	343	679	FAIL
SILICONE	20.0	1067	6	1061	FAIL

NEITHER CONTAMINANT FLUORESCED UNDER BLACK LIGHT EXPOSURE. OSEE DATA TAKEN ON GENERATION II SYSTEM. COATING LEVEL DETERMINED BY MEASURING THE WEIGHT CHANGE OF AN ALUMINUM WITNESS FOIL SPRAYED ALONG WITH THE PANEL.

AC93TIMK

TABLE XI
WET TAPE TEST vs TENSILE ADHESION
(2219-T87 ALUMINUM/ET DESOTO PRIMER: ENVIRONMENTAL
EXPOSURE/CONTAMINATION STUDY)

<u>PANEL</u>	<u>SURFACE HISTORY</u>	<u>WET TAPE ADHESION TEST *</u>	<u>TENSILE ADHESION (STRESS @ PEAK) (psi)**</u> <u>MEAN/STD.DEV</u>	<u>MODE OF FAILURE</u> <u>***</u>
EA1	48HRS/65F/20RH	PASS	1406/100	40% P/M 60% P/A
EACA11	+ 1mg/ft2 Kaydol	PASS	1458/119	40% P/M 60% P/A
EACA21	+ 5 mg/ft2 Kaydol	PASS	1439/256	40% P/M 60% P/A
EACA31	+ 10 mg/ft2 Kaydol	PASS	1315/350	40% P/M 60% P/A
EACB11	+ 1 mg/ft2 Silicone	PASS	845/276	90% P/M 10% P/A
EACB21	+ 5 mg/ft2 Silicone	PASS	206/40	100% P/M
EACB31	+ 10 mg/ft2 Silicone	PASS	143/51	100% P/M
EA2	96HRS/65F/20RH	PASS	1697/274	20% P/M 80% P/A
EACA12	+ 1mg/ft2 Kaydol	PASS	1754/290	60% P/A 40% P/M
EACA22	+ 5 mg/ft2 Kaydol	PASS	1527/524	80% P/A 20% P/M
EACA32	+ 10 mg/ft2 Kaydol	FAIL	1577/320	60% P/A 40% P/M
EACB12	+ 1 mg/ft2 Silicone	PASS	796/213	90% P/M 10% P/A
EACB22	+ 5 mg/ft2 Silicone	FAIL	165/31	100% P/M
EACB32	+ 10 mg/ft2 Silicone	PASS	130/66	100% P/M
EA3	48HRS/65F/60RH	PASS	1083/352	90% P/A 10% P/M
EACA13	+ 1mg/ft2 Kaydol	PASS	1504/308	30% P/M 70% P/A
EACA23	+ 5 mg/ft2 Kaydol	FAIL	1266/218	20% P/M 80% P/A
EACA33	+ 10 mg/ft2 Kaydol	PASS	988/87	40% P/M 60% P/A
EACB13	+ 1 mg/ft2 Silicone	PASS	969/318	50% P/M 50% P/A
EACB23	+ 5 mg/ft2 Silicone	PASS	124/31	100% P/M
EACB33	+ 10 mg/ft2 Silicone	FAIL	121/49	100% P/M

* Performed per Martin Marietta procedure MPI PI-3003-1

**Tensile adhesion measured by bonding 1.25" diameter steel buttons to primed panels using Versilok 201, then pulling with an Instron machine (@ 0.05"/min)

***P=PRIMER, M=METAL (2219-T87 ALUMINUM), A=ADHESIVE

TABLE XI (cont.)

<u>PANEL</u>	<u>SURFACE HISTORY</u>	<u>WET TAPE ADHESION TEST *</u>	<u>TENSILE ADHESION (STRESS @ PEAK) (psi)** MEAN/STD.DEV</u>	<u>MODE OF FAILURE ***</u>
EA4	96HRS/65F/60RH	PASS	1259/268	50% P/M 50% P/A
EACA14	+ 1mg/ft2 Kaydol	PASS	1387/258	70% P/M 30% P/A
EACA24	+ 5 mg/ft2 Kaydol	PASS	1126/326	70% P/M 30% P/A
EACA34	+ 10 mg/ft2 Kaydol	PASS	1364/280	70% P/M 30% P/A
EACB14	+ 1 mg/ft2 Silicone	PASS	500/165	100% P/M
EACB24	+ 5 mg/ft2 Silicone	PASS	90/32	100% P/M
EACB34	+ 10 mg/ft2 Silicone	PASS	96/28	100% P/M
EA5	48HRS/100F/20RH	PASS	1642/174	90% P/A 10% P/M
EACA15	+ 1mg/ft2 Kaydol	PASS	1416/266	95% P/A 5% P/M
EACA25	+ 5 mg/ft2 Kaydol	PASS	1132/310	95% P/A 5% P/M
EACA35	+ 10 mg/ft2 Kaydol	PASS	1589/269	90% P/A 10% P/M
EACB15	+ 1 mg/ft2 Silicone	PASS	1321/294	70% P/M 30% P/A
EACB25	+ 5 mg/ft2 Silicone	PASS	136/58	100% P/M
EACB35	+ 10 mg/ft2 Silicone	FAIL	68/22	100% P/M
REA6	96HRS/100F/20RH	PASS	1683/321	30% P/M 70% P/A
RA16	+ 1mg/ft2 Kaydol	PASS	1624/396	10% P/M 90% P/A
RA26	+ 5 mg/ft2 Kaydol	PASS	1580/258	20% P/M 80% P/A
RA36	+ 10 mg/ft2 Kaydol	PASS	1550/322	10% P/M 90% P/A
RB16	+ 1 mg/ft2 Silicone	PASS	1091/434	90% P/M 10% P/A
RB26	+ 5 mg/ft2 Silicone	PASS	316/170	100% P/M
RB36	+ 10 mg/ft2 Silicone	FAIL	521/167	100% P/M

* Performed per Martin Marietta procedure MPI PI-3003-1

**Tensile adhesion measured by bonding 1.25" diameter steel buttons to primed panels using Versilok 201, then pulling with an Instron machine (@ 0.05"/min.)

***P=PRIMER, M=METAL (2219-T87 ALUMINUM), A=ADHESIVE

TABLE XI (cont.)

<u>PANEL</u>	<u>SURFACE HISTORY</u>	<u>WET TAPE ADHESION TEST *</u>	<u>TENSILE ADHESION (STRESS @ PEAK) (psi)** MEAN/STD. DEV</u>	<u>MODE OF FAILURE ***</u>
EA7	48HRS/100F/60RH	PASS	1218/193	30% P/M 70% P/A
EACA17	+ 1mg/ft2 Kaydol	PASS	1435/329	80% P/A 20% P/M
EACA27	+ 5 mg/ft Kaydol	PASS	1199/306	50% P/A 50% P/M
EACA37	+ 10 mg/ft2 Kaydol	PASS	1382/352	70% P/A 30% P/M
EACB17	+ 1 mg/ft2 Silicone	PASS	809/195	90% P/M 10% P/A
EACB27	+ 5 mg/ft2 Silicone	FAIL	254/58	100% P/M
EACB37	+ 10 mg/ft2 Silicone	PASS	177/77	100% P/M
EA8	96HRS/100F/60RH	PASS	1182/106	100% P/A
EACA18	+ 1mg/ft2 Kaydol	PASS	1508/227	90% P/A 10% P/M
EACA28	+ 5 mg/ft2 Kaydol	PASS	1378/230	90% P/A 10% P/M
EACA38	+ 10 mg/ft2 Kaydol	PASS	1298/280	90% P/A 10% P/M
EACB18	+ 1 mg/ft2 Silicone	PASS	1257/104	50% P/M 50% P/A
EACB28	+ 5 mg/ft2 Silicone	FAIL	169/40	100% P/M
EACB38	+ 10 mg/ft2 Silicone	FAIL	137/52	100% P/M

* Performed per Martin Marietta procedure MPI PI-3003-1

**Tensile adhesion measured by bonding 1.25" diameter steel buttons to primed panels using Versilok 201, then pulling with an Instron machine (@ 0.05"/min.)

***P=PRIMER, M=METAL (2219-T87 ALUMINUM), A=ADHESIVE

TABLE XII
PENCIL HARDNESS VS. CONTAMINATION LEVEL
2219-T87 ALUMINUM ENVIRONMENTAL EXPOSURE/
CONTAMINATION STUDY

PANEL SET	Baseline (Uncoated panel)	1 mg/ft ² Kaydol	5 mg/ft ² Kaydol	10 mg/ft ² Kaydol	1 mg/ft ² CRC Si	5 mg/ft ² CRC Si	10 mg/ft ² CRC Si
EA1	5H	5H	5H	5H	5H	F	F
EA2	6H	6H	6H	5H	5H	2H	F
EA3	5H	5H	5H	3H	3H	F	F
EA4	5H	5H	5H	5H	5H	H	HB
EA5	6H	6H	6H	5H	5H	F	F
EA6	5H	6H	5H	6H	5H	3H	2H
EA7	6H	6H	6H	6H	5H	2H	H
EA8	6H	6H	6H	6H	6H	2H	H

2219-T87 aluminum panels exposed to environmental conditions typical for External Tank production facility (See Taguchi L8 Array-2219-T87 Environmental Exposure/Contamination Study). Panels coated with Kaydol or CRC Silicone using a Binks Wren airbrush. ET Desota primer applied in a separate airbrush facility.

Pencil Hardness test performed according to specification ASTM-D-3363. Test performed with a set of calibrated drawing leads (A.W. Faber-Castell 9000) meeting the following scale of hardness (with hardness increasing left to right):

6B-5B-4B-3B-2B-B-HB-F-H-2H-3H-4H-5H-6H

The difference between 2 adjacent leads is considered one unit of hardness

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TABLE XIII
FACTOR ANALYSIS OF 2219-T87 ALUMINUM
TAGUCHI ARRAY WITH ENVIRONMENTAL
CHAMBER OSEE DELTA AS RESPONSE

<u>FACTORS</u>								RESPONSE
<u>RUN</u>	<u>A</u> <u>TEMP</u>	<u>B</u> <u>R.H.</u>	<u>AXB</u> <u>TEMP</u> <u>X</u> <u>R.H.</u>	<u>C</u> <u>TIME</u>	<u>AXC</u> <u>TEMP</u> <u>X</u> <u>TIME</u>	<u>BXC</u> <u>R.H. X</u> <u>TIME</u>	<u>AXBXC</u> <u>TEMP</u> <u>X R.H.</u> <u>X</u> <u>TIME</u>	
1	65	20	1	48 HRS	1	1	1	72
2	65	20	1	96 HRS	2	2	2	230
3	65	60	2	48 HRS	1	2	2	120
4	65	60	2	96 HRS	2	1	1	180
5	100	20	2	48 HRS	2	1	2	570
6	100	20	2	96 HRS	1	2	1	560
7	100	60	1	48 HRS	2	2	1	590
8	100	60	1	96 HRS	1	1	2	830
TOTAL								3152
AVG.								394.00
STD. DEV.								277.36
TOTAL SUM SQUARES								538496
SUM OF SQUARES FOR EACH VARIABLE	474338	10368	10658	25088	18	2888	15138	
% CONTRI-BUTION TO VARIATION	88.09	1.93	1.98	4.66	0.00	0.54	2.81	TOTAL=100

• OSEE DELTA TAKEN FROM READINGS INSIDE THE ENVIRONMENTAL CHAMBER (GENERATION II SYSTEM).

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TABLE XIV
FACTOR ANALYSIS OF 2219-T87 ALUMINUM
TAGUCHI ARRAY WITH TENSILE ADHESION AS
RESPONSE

<u>FACTORS</u>								RESPONSE
<u>RUN</u>	<u>A</u> <u>TEMP</u>	<u>B</u> <u>R.H.</u>	<u>AXB</u> <u>TEMP</u> <u>X</u> <u>R.H.</u>	<u>C</u> <u>TIME</u>	<u>AXC</u> <u>TEMP</u> <u>X</u> <u>TIME</u>	<u>BXC</u> <u>R.H. X</u> <u>TIME</u>	<u>AXBXC</u> <u>TEMP</u> <u>X R.H.</u> <u>X</u> <u>TIME</u>	<u>TENSILE</u> <u>ADHESION</u> <u>(psi)*</u>
1	65	20	1	48 HRS	1	1	1	1406
2	65	20	1	96 HRS	2	2	2	1697
3	65	60	2	48 HRS	1	2	2	1083
4	65	60	2	96 HRS	2	1	1	1259
5	100	20	2	48 HRS	2	1	2	1642
6	100	20	2	96 HRS	1	2	1	1683
7	100	60	1	48 HRS	2	2	1	1218
8	100	60	1	96 HRS	1	1	2	1182
TOTAL								11170
AVG.								1396
STD. DEV.								247.21
TOTAL SUM SQUARES								427804
SUM OF SQUARES FOR EACH VARIABLE	9800	355345	3362	27848	26681	4608	181	
% CONTRI-BUTION TO VARIATION	2.29	83.06	0.79	6.51	6.24	1.08	0.04	TOTAL=100

* Panels primed with ET Desoto primer. Tensile adhesion measured by bonding 1.25" diameter steel buttons to primed panels using Versilok 201, then pulling with an Instron machine (@ 0.05"/min.)

** All panels exhibited varying degrees of primer to Versilok failure (50-100%).

TABLE XV
FACTOR ANALYSIS OF 2219-T87 ALUMINUM
TAGUCHI ARRAY WITH CRC SILICONE AS THE
CONTAMINANT

<u>RUN</u>	<u>TEMPERATURE</u> <u>(DEGREES</u> <u>F)</u>	<u>RELATIVE</u> <u>HUMIDITY</u> <u>(%)</u>	<u>EXPOSURE</u> <u>TIME</u> <u>(HOURS)</u>	<u>CONTAMINATION</u> <u>LEVEL (MG/FT2)</u>	<u>ENVIRON-</u> <u>MENTAL</u> <u>CHAMBER</u> <u>DELTA</u> <u>OSEE</u>	<u>TENSILE</u> <u>ADHE-</u> <u>SION</u> <u>(psi)*</u>
1	65	20	48	0	0	1406
1	65	20	48	1	321	845
1	65	20	48	5	839	206
1	65	20	48	10	1012	143
2	65	20	96	0	0	1697
2	65	20	96	1	496	796
2	65	20	96	5	589	165
2	65	20	96	10	785	130
3	65	60	48	0	0	1083
3	65	60	48	1	370	969
3	65	60	48	5	895	124
3	65	60	48	10	950	121
4	65	60	96	0	0	1259
4	65	60	96	1	341	500
4	65	60	96	5	452	90
4	65	60	96	10	651	96
5	100	20	48	0	0	1642
5	100	20	48	1	195	1321
5	100	20	48	5	673	136
5	100	20	48	10	931	68
6	100	20	96	0	0	1683
6	100	20	96	1	638	1091
6	100	20	96	5	897	316
6	100	20	96	10	980	521
7	100	60	48	0	0	1218
7	100	60	48	1	317	809
7	100	60	48	5	706	254
7	100	60	48	10	870	177
8	100	60	96	0	0	1182
8	100	60	96	1	155	1257
8	100	60	96	5	570	169
8	100	60	96	10	832	137

* After environmental exposure/contamination, panels primed with ET Desoto primer. Tensile adhesion measured by bonding 1.25" diameter steel buttons to primed panels using Versilok 201, then pulling with an Instron machine (@ 0.05"/min.)

TABLE XVI
FACTOR ANALYSIS OF 2219-T87 ALUMINUM
TAGUCHI ARRAY WITH KAYDOL AS THE
CONTAMINANT

<u>RUN</u>	<u>TEMPERA- TURE</u> (<u>DEGREES</u> <u>F</u>)	<u>RELATIVE</u> <u>HUMIDITY</u> (<u>%</u>)	<u>EXPOSURE</u> <u>TIME</u> (<u>HOURS</u>)	<u>CONTAMINATION</u> <u>LEVEL (MG/FT2)</u>	<u>ENVIRON- MENTAL</u> <u>CHAMBER</u> <u>DELTA</u> <u>OSEE</u>	<u>TENSILE</u> <u>ADHE- SION</u> (<u>psi</u>)*
1	65	20	48	0	0	1406
1	65	20	48	1	75	1458
1	65	20	48	5	384	1439
1	65	20	48	10	840	1315
2	65	20	96	0	0	1697
2	65	20	96	1	200	1754
2	65	20	96	5	567	1527
2	65	20	96	10	699	1577
3	65	60	48	0	0	1083
3	65	60	48	1	93	1504
3	65	60	48	5	488	1266
3	65	60	48	10	759	988
4	65	60	96	0	0	1259
4	65	60	96	1	56	1387
4	65	60	96	5	289	1126
4	65	60	96	10	562	1364
5	100	20	48	0	0	1642
5	100	20	48	1	37	1416
5	100	20	48	5	240	1132
5	100	20	48	10	610	1589
6	100	20	96	0	0	1683
6	100	20	96	1	297	1624
6	100	20	96	5	587	1580
6	100	20	96	10	950	1550
7	100	60	48	0	0	1218
7	100	60	48	1	120	1435
7	100	60	48	5	409	1199
7	100	60	48	10	743	1382
8	100	60	96	0	0	1182
8	100	60	96	1	98	1508
8	100	60	96	5	471	1378
8	100	60	96	10	882	1298

* After environmental exposure/contamination, panels primed with ET Desoto primer. Tensile adhesion measured by bonding 1.25" diameter steel buttons to primed panels using Versilok 201, then pulling with an Instron machine (@ 0.05"/min.)

TABLE XVII
FACTOR ANALYSIS OF 2219-T87 ALUMINUM TAGUCHI ARRAY WITH
KAYDOL AS THE CONTAMINANT AND TENSILE ADHESION AS
RESPONSE

<u>RUN</u>	<u>I</u>	<u>H</u>	<u>E</u>	<u>C</u>	<u>T X H</u>	<u>T X E</u>	<u>T X C</u>	<u>H X E</u>	<u>H X C</u>	<u>E X C</u>	<u>T X H X E X C</u>
SUM OF SQUARES FOR EACH VARIABLE	13861	454105	127765	30870	10513	6105	13850	42632	3381	318	480875
% CONTRI- BUTION TO VARIATION	1.18	38.99	10.97	2.65	0.90	0.52	1.19	3.66	0.29	0.03	39.59

T=TEMPERATURE (F) H=RELATIVE HUMIDITY (%) E=EXPOSURE TIME (HOURS) C=CONTAMINATION (MG/FT²)

Panels primed with ET Desoto primer. Tensile adhesion measured by bonding 1.25" diameter steel buttons to primed panels using Versilok 201, then pulling with an Instron machine (@ 0.05"/min.).

All panels exhibited predominantly primer to Versilok failure (50-100%), therefore actual primer to aluminum adhesion is higher than reported values. Statistical analysis was performed on the measured tensile adhesion strengths.

TABLE XVIII
FACTOR ANALYSIS OF 2219-T87 ALUMINUM TAGUCHI ARRAY WITH
CRC SILICONE AS THE CONTAMINANT AND TENSILE ADHESION AS
RESPONSE

<u>RUN</u>	<u>I</u>	<u>H</u>	<u>E</u>	<u>C</u>	<u>T X H</u>	<u>T X E</u>	<u>T X C</u>	<u>H X E</u>	<u>H X C</u>	<u>E X C</u>	<u>T X H X</u> <u>E X C</u>
SUM OF SQUARES FOR EACH VARIABLE	17272 5	231370	10047	6685072	5751	25032	12161	15182	75661	3314	2581137
% CONTRI- BUTION TO VARIATION	1.76	2.35	0.10	68.13	0.06	0.25	0.12	0.15	0.77	0.03	26.26

T=TEMPERATURE (F) H=RELATIVE HUMIDITY (%) E=EXPOSURE TIME (HOURS) C=CONTAMINATION (MG/FT²)

Panels primed with ET Desoto primer. Tensile adhesion measured by bonding 1.25" diameter steel buttons to primed panels using Versilok 201, then pulling with an Instron machine (@ 0.05"/min.).

All silicone contaminated panels exhibited predominantly primer to aluminum failure (50-100%).

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TABLE XIX
FACTOR ANALYSIS OF 2219-T87 ALUMINUM TAGUCHI ARRAY WITH
KAYDOL AS THE CONTAMINANT AND OSEE DELTA AS RESPONSE

<u>RUN</u>	<u>I</u>	<u>H</u>	<u>E</u>	<u>C</u>	<u>T X H</u>	<u>T X E</u>	<u>T X C</u>	<u>H X E</u>	<u>H X C</u>	<u>E X C</u>	<u>I X H X E X C</u>
SUM OF SQUARES FOR EACH VARIABLE	5832	8321	23113	2726911	8450	60552	4813	58482	198	137	130287
% CONTRI- BUTION TO VARIATION	0.19	0.27	0.76	90.08	0.28	2.00	0.16	1.93	0.01	0.01	4.30

T=TEMPERATURE (F) H=RELATIVE HUMIDITY (%) E=EXPOSURE TIME (HOURS) C=CONTAMINATION (MG/FT²)

OSEE measurements made using the laboratory environment (generation II) system.

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TABLE XX
FACTOR ANALYSIS OF 2219-T87 ALUMINUM TAGUCHI ARRAY WITH
CRC SILICONE AS THE CONTAMINANT AND OSEE AS RESPONSE

<u>RUN</u>	<u>I</u>	<u>H</u>	<u>E</u>	<u>C</u>	<u>I X H</u>	<u>I X E</u>	<u>I X C</u>	<u>H X E</u>	<u>H X C</u>	<u>E X C</u>	<u>I X H X E X C</u>
SUM OF SQUARES FOR EACH VARIABLE	124	48594	15008	3098168	7230	65975	8308	72295	3974	49921	710279
% CONTRI- BUTION TO VARIATION	.01	1.19	0.34	75.93	0.18	1.62	0.20	1.77	0.09	1.22	17.42

T=TEMPERATURE (F) H=RELATIVE HUMIDITY (%) E=EXPOSURE TIME (HOURS) C=CONTAMINATION (MG/FT²)

OSEE measurements made using the laboratory environment (generation II) system.

TABLE XXI: OXIDE THICKNESS MEASUREMENTS

Panel Set	Exposure Conditions	Aluminum Barrier Oxide Measurement (1) (Applied Voltage=3V)	Total Oxide Thickness Measurement (2)				Aluminum Hydroxide Thickness (3) (Angstroms)
		Oxide Thickness (Angstroms)	Optical Constant	Absorption Constant	Oxide Thickness (Angstroms)		
1	65F/20%RH/48HRS	18.7	1.600	0	192	173.3	
2	65F/20%RH/96HRS	18.7	1.600	0	97 (73-165)	78.3	
3	65F/60%RH/48HRS	17.5	1.56	0	95	77.5	
4	65F/60%RH/96HRS	14	1.62	0	189	175	
5	100F/20%RH/48HRS	14	1.600	0	132 (114-177)	118	
6	100F/20%RH/96HRS	14	1.750	0.1	162	148	
7	100F/60%RH/48HRS	11.7	5.144	0	142	130.3	
8	100F/60%RH/96HRS	9.3	1.600	0	200 (165-219)	190.7	

(1) Barrier oxide (aluminum oxide) thickness determined by immersing exposed panel and clean reference panel in a solution of 3% tartaric acid adjusted to pH=5.5 with ammonium hydroxide, applying a voltage slowly across the panels, and plotting voltage vs. current flow. The barrier oxide thickness is 14 times the highest voltage that does not produce a pronounced increase in current flow.

(2) Total oxide (aluminum oxide plus aluminum hydroxide) thickness determined by ellipsometry.

(3) Calculated by subtracting Barrier Oxide Thickness from Total Oxide Thickness.

TABLE XXII

TAGUCHI DESIGN FOR HP9-4-30/ EPDM BONDLINE ENVIRONMENTAL TESTS

FACTORS

LEVELS	<u>A</u> TEMP		<u>B</u> REL. HUMIDITY		<u>C</u> EXPOSURE TIME	
	$T_1 = 70^\circ\text{F}$	$T_2 = 80^\circ\text{F}$	$\text{RH}_1 = 40\%$	$\text{RH}_2 = 75\%$	$t_1 = 4 \text{ Hrs}$	$t_2 = 48 \text{ Hrs}$
					Followed by 24 Hrs. at 75F/55% RH	

TAGUCHI L8 ORTHOGONAL ARRAY (RESOLUTION 4, ALL MAIN EFFECTS / INTERACTIONS CAN BE ESTIMATED)

RUN	<u>FACTORS</u>							<u>RESPONSES</u>			
	1 A	2 B	3 AxB	4 C	5 AxC	6 BxC	7 AxBxC	OSEE	OSEE Δ	PS Av. 7	TS Av. 6
1	1	1	1	1	1	1	1	0 ₁	D ₁	P ₁	T ₁
2	1	1	1	2	2	2	2	0 ₂	D ₂	P ₂	T ₂
3	1	2	2	1	1	2	2	0 ₃	D ₃	P ₃	T ₃
4	1	2	2	2	2	1	1	0 ₄	D ₄	P ₄	T ₄
5	2	1	2	1	2	1	2	0 ₅	D ₅	P ₅	T ₅
6	2	1	2	2	1	2	1	0 ₆	D ₆	P ₆	T ₆
7	2	2	1	1	2	2	1	0 ₇	D ₇	P ₇	T ₇
8	2	2	1	2	1	1	2	0 ₈	D ₈	P ₈	T ₈

Four hour to 48 hour exposure time simulates time delay between aqueous cleaning and move into airlock facility. The subsequent 24 hour exposure at 55%RH simulates time delay prior to Chemlok application.

TABLE XXIII
CONTRACT NAS8-39244
TAGUCHI OUTER ARRAY DESIGN FOR HP9-4-30 STEEL
CONTAMINATION TESTS

LEVELS

Contaminant: A= Kaydol Oil B= HD2 Grease C= CRC Silicone	Concentration (mg/ft ²):	For A & B: 1= 25 2=200 For C: 1= 2 2=20
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RUN	CONTAMINATION			RESPONSES					
	A	B	C	OSEE Δ			P/TS		
1	1	1	1	D _{1A}	D _{1B}	D _{1C}	T _{1A}	T _{1B}	T _{1C}
2	2	2	2	D _{2A}	D _{2B}	D _{2C}	T _{2A}	T _{2B}	T _{2C}

***T.A.-Tape adhesion test**

TABLE XXIV: EPDM 44010 INSULATION LAY-UP AND VACUUM BAGGING PROCEDURE

SCOPE

This procedure shall provide instructions for insulation lay-up and vacuum bagging of test panels designed to provide adhesive bond data.

MATERIALS AND EQUIPMENT

The following list describes equipment and materials required for this operation:

- Kevlar filled EPDM insulation, specification 44010
- Missile grade air
- Gloves, nitrile and rubber
- Rymple cloth
- Breather cloth, Richmond 3000 or equivalent
- Thermocouples, type "J"
- Plastic film, nylon, Wrighton 8400 or equivalent
- Bleeder cloth, Lease "C"
- Tape, vacuum sealant
- Tape, Teflon
- HP9-4-30 steel panels coated with Chemloks 205/236A
- D6AC steel beveled buttons, 1.25" diameter, coated with Chemloks 205/236A
- Teflon coated template
- Metal scrim, coated with Chemloks 205/236A
- Stanley razor blade knives

TEST PANEL INSULATION LAY-UP

NOTE: Test panels shall be prepared per Figure I

1. Place the Teflon coated aluminum masking frame over the insulation and trace out two plies using a marking pen Cut the insulation such that the fiber direction is the same as the peel direction
2. Carefully cut out each ply and place it on a clean working surface
3. Place the Teflon coated masking frame over metal scrim and trace out 1 piece using a marking pen
4. Carefully cut out each piece and place it on a clean working surface

5. Place a 3" wide strip of Teflon tape along the length of the test panel in the peel area
6. Place the Teflon coated masking frame over the test panel
7. Remove the polyethylene off the first insulation ply and place the ply onto the test panel using the masking frame as a guide. The side of the insulation which had the polyethylene is placed toward the panel
8. Place one piece of metal scrim on top of the first insulation ply
9. Place "J" type thermocouple on top of metal scrim
10. Repeat step 8 for the second ply
11. Cut one 1.25" diameter circle of the insulation for each of the 6 circles on the Teflon coated masking frame
12. Remove the polyethylene backing and place one piece of insulation in each slot in the masking frame
13. Place 1 Chemlok coated beveled steel button on top of each circular insulation specimen

VACUUM BAGGING PROCEDURE

1. Obtain a clean autoclave plate for vacuum bagging test panels
2. Apply plastic film (Airtech A 4000 R/non-perforated, or equivalent) to the plate surface in the area which the panels will be placed on the plate. Tape the film down with Teflon tape

NOTE: Insure that the vacuum port on the autoclave plate is not covered with the film or tape material

3. Place test panels on the plastic film making sure the plates are pushed together
4. Cut and roll up pieces of breather cloth to go around the perimeter of the test panels. Tape these down to the autoclave plate and along the outside edge of the test panels
5. Place a small aluminum plate wrapped in breather cloth over the autoclave plate vacuum port
6. Cut one piece of bleeder cloth to cover the test panels. Make sure the smooth side of the bleeder cloth is against the insulation

7. Cut one piece of breather cloth large enough to cover the test panels
8. Place tacky tape around the outside perimeter of the autoclave plate surface
9. Lay thermocouple wires from the panels over the tacky tape
10. Cut a piece of nylon film vacuum bag material approximately one foot larger (in both dimensions) than the autoclave plate
11. Apply tacky tape to the vacuum bag film along the outer perimeter
12. Punch holes in any tape or breather cloth positioned over the autoclave vacuum port
13. Mate the vacuum bag film tacky tape and autoclave plate tacky tape, making sure tape surfaces are firmly pressed together
14. Make creases in vacuum bag on all sides of the plate. Align vacuum bag creases with spaces between plates and the outer plate edges so that the bag material will not be forced down onto panel edges
15. Attach a vacuum hose between the autoclave plate and vacuum source
16. Allow vacuum bag to pull down while listening for leaks. If leaks are detected, firmly press tacky tape together in the leak area. A minimum vacuum level of 25 inches of Hg should be obtained. If proper vacuum level is not obtained, remove the vacuum bag, reapply tacky tape and repeat bagging procedure
17. Allow vacuum source to pump on vacuum bag for approximately 1 hour, then disconnect the bag from the vacuum source. Connect a vacuum gauge to the autoclave plate and insure that the vacuum level does not decay more than 1 to 1-1/2 inches of Hg over a 15 minute period
18. Move bagged panels to autoclave area for vulcanization

TABLE XXV
EFFECT OF ENVIRONMENTAL EXPOSURE/CONTAMINATION
ON HP9-4-30 STEEL OSEE RESPONSE/EPDM ADHESION

TEST SPECIMEN FLOW:
Taguchi Run___/___°F/___%RH/___HRS

- Note:** 1. Approved gloves to be used in the handling of panels.
2. A log will be kept for each set of panels in which all pertinent data shall be recorded.

Date:_____

- ____1. *Verify Test & Witness Panel Configurations & Serial Numbers*
-Size:
1 ea. Test Panel - 8"x12"x1/8"
-Serial Numbers:
Environmental Test Panel ____
Contamination Test Panels:____
- ____2. *Cosmetic Zirclean Grit Blast*
-90° impingement
- 80 psi nozzle pressure
- Target roughness 70-120 μ in
- ____3. *Aqueous Cleaning (10% Brulin 815 GD) DATE:*
- Preheat 5-gal bath to 150° \pm 5°F
- Induce agitation to 4% by volume per minute
- Completely immerse panels in solution for 1 1/2 hours
- Rinse panels with 140°F DI water, (approx 800 ml per 8 x 12" panel).
- Promptly blow dry using missile grade air
- Nitrogen purge and seal in Capran.

Panel:___	Time in:___	Time out:___	Bagged:___
Panel:___	Time in:___	Time out:___	Bagged:___
Panel:___	Time in:___	Time out:___	Bagged:___
Panel:___	Time in:___	Time out:___	Bagged:___
Panel:___	Time in:___	Time out:___	Bagged:___
Panel:___	Time in:___	Time out:___	Bagged:___
Panel:___	Time in:___	Time out:___	Bagged:___

4. Prepare Environmental Chamber & OSEE System (w/ controlled z-axis scanning). NOTE: OSEE values not corrected for grains of moisture

- Run OSEE System: Check w/ Calibration surface
 - Calibration Std.: Type_ID#__
 - OSEE Value: mean__std.dev__
- Set Controls for Chamber
 - Temperature__°F/R.H.__%/Exposure Duration__
- Record OSEE, Chamber Settings & Barometric Pressure in Log Book
- Scan of Calibration Std. in chamber
 - Type_ID#__; mean__std.dev.__

-Initial scan of panel after cleaning (OSEE generation II, Table I Room 111)

Panel: Time: Ra: mean: std.dev.: Placed in Chamber:

Panel:___ Time:___ Ra:___ mean:___ std.dev:___ Placed in Chamber:___
 Panel:___ Time:___ Ra:___ mean:___ std.dev:___ Placed in Chamber:___

Panel:___ Time:___ Ra:___ mean:___ std.dev.:___ Placed in Chamber:___
Panel:___ Time:___ Ra:___ mean:___ std.dev.:___ Placed in Chamber:___

Panel:___Time:___Ra:___mean:___std.dev.:___Placed in Chamber:___
Panel:___Time:___Ra:___mean:___std.dev.:___Placed in Chamber:___

Panel:___Time:___Ra:___mean:___std.dev.:___Placed in Chamber___

Panel: ___ Time: ___ Ra: ___ mean: ___ std. dev.: ___ Placed in Chamber: ___
 Panel: ___ Time: ___ Ra: ___ mean: ___ std. dev.: ___ Placed in Chamber: ___

Panel:___Time:___Ra:___mean ___std.dev.:___Placed in Chamber:___

5. *Initiate Environmental Exposure*

- Close Chamber Door and Log Time. Conditions: TEST START**
TIME_

6. OSEE Measurements

- _____6. *OSEE Measurements*
 -Set System to Repeat Scan Every__minutes for__hrs, then every__
 hours until end of test-phase I. Reset chamber for Airlock Simulation Phase--24 hrs at
 75F/55%RH, set scans for every__ Hrs to end of test.

7. Termination of Environmental Exposure DATE: _____

- Remove Test Panel from Chamber and Log Time_____
- OSEE scan (OSEE Generation II, Table I, Room 111):

Panel: __ Time: __ mean: __ std.dev.: __

Panel: __ Time: __ mean: __ std.dev.: __

Panel: __ Time: __ mean: __ std.dev: __

Panel: __ Time: __ mean: __ std.dev.

Panel: __ Time: __ mean: __ std.dev.: __

Panel: __ Time: __ mean: __ std.dev.: __

Panel: __ Time: __ mean: __ std.dev.: __

_____8. Contaminant panel (if applicable): Scan on OSEE generation II

- Panel _____

-Contaminant type _____

-Desired concentration _____mg/ft²

-Prepare witness aluminum foil

-Spray uniform coat of contaminant over test panel and witness foil

-Allow 3 minutes for carrier solvent to evaporate

-Concentration in mg/ft² _____

-OSEE scan after contamination: TIME/TEMP/RH _____

mean=_____ std.dev.=_____

- Panel _____

-Contaminant type _____

-Desired concentration _____mg/ft²

-Prepare witness aluminum foil

-Spray uniform coat of contaminant over test panel and witness foil

-Allow 3 minutes for carrier solvent to evaporate

-Concentration in mg/ft² _____

-OSEE scan after contamination: TIME/TEMP/RH _____

mean=_____ std.dev.=_____

- Panel _____

-Contaminant type _____

-Desired concentration _____mg/ft²

-Prepare witness aluminum foil

-Spray uniform coat of contaminant over test panel and witness foil

-Allow 3 minutes for carrier solvent to evaporate

-Concentration in mg/ft² _____

-OSEE scan after contamination: TIME/TEMP/RH _____

mean=_____ std.dev.=_____

- Panel _____

-Contaminant type _____

-Desired concentration _____mg/ft²

-Prepare witness aluminum foil

-Spray uniform coat of contaminant over test panel and witness foil

-Allow 3 minutes for carrier solvent to evaporate

-Concentration in mg/ft² _____

-OSEE scan after contamination: TIME/TEMP/RH _____

mean=_____ std.dev.=_____

- Panel _____

-Contaminant type _____

-Desired concentration _____mg/ft²

- ____10. -*Insulation Lay-Up*
- Lay down teflon tape to form 3" pull tab
- Lay up EPDM (W/ Scrim steel reinforcement and thermocouples; scrim to be grit blasted and Chemloked along with panels)
 -Thickness of each layer 0.1 INCH
 - # of layers - per peel 2
 per tensile 1
- Lay up bleeder cloth/breather cloth
- Vacuum bag and seal bond test plate; pressure____(**)
- ____11. -*Cure In Autoclave According to pre-approved procedure(**)*
- ____12. - *Prepare Test Specimens According to Layout on Bond Specimen Drawing*
- Take Hardness Measurement (Shore A) and Record in Log Book
 mean__min__max__
- Cut 1" peel specimens w/ Exacto knife
- Label Each specimen with test plate serial number and specimen number.(**)
- ____13. - *Test Specimens*
- Peel Angle 45°/ Peel Rate 2 in/min/ Tensile Loading Rate 0.5in/min
-Record Results in Log Book for Each Specimen along w/ Temp/RH
- Enter All Data in Computer Data Base
- (**) Hold point- sequence can be interrupted w/o interfering w/ test design.

**TABLE XXVI: CONDUCTIVITY/pH OF TURCO 3878 LF-NC SOLUTIONS (20%)
USED TO CLEAN HP9-4-30 STEEL**

SAMPLE SOURCE*	PANELS PROVIDED BY	MACHINED FLAT @ MISFC	VAPOR-DEGREASED AFTER MACHINING	GRIT BLASTED	DISCOLORED AFTER AQUEOUS CLEANING	CONDUCTIVITY.ms (mmhos)	pH
Fresh, unused solution (Turco taken from 25-gal drum in Bldg. 4707)	ACE	N/A	N/A	N/A	N/A	3.87	8.5
5-gal tank	Aerojet	N	N	Y	N	3.76	8.5
5-gal tank	ACE	Y	Y	Y	N	4.39	8.5
5-gal tank	ACE	N	N	Y	N	4.1	
5-gal. tank, w/ 2% cutting oil	ACE	Y	Y	Y	N	4.2	8.6
5-gal.tank w/ 4% cutting oil	ACE	Y	Y	Y	N	4.3	8.7
5-gal. tank*	ACE	Y	N	Y	Y	12.1	9.0
60-gal. tank	ACE	Y	N	Y	Y	10.85	8.6
60-gal. tank (freshly charged)	ACE	Y	N	Y	Y	9.95	8.8
60-gal. tank (2nd fresh charge)	Aerojet	N	N	Y	N	10.2	8.8
5-gal. tank (taken from 2nd fresh charge 60-gal.)	ACE	Y	Y	Y	N	-	-
5-gal. tank (taken from 2nd fresh charge 60-gal.)	ACE	N	N	N	N	-	-
Fresh unused solution (Turco taken from new 55-gal. drum in Bldg. 4760)	N/A	N/A	N/A	N/A	N/A	11.41	8.5

*This solution contained a significant amount of HD2 grease from previous experiments.

** Turco taken from 25-gal. drum in Bldg. 4707 unless otherwise noted.

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TABLE XXVII
ICP ANALYSIS OF FRESH VS. TAINTED TURCO
SOLUTIONS AS PERFORMED BY
SOUTHEASTERN ANALYTICAL SERVICES

<u>ELEMENT</u>	<u>FRESH TURCO SOLUTION (mg/L)*</u>	<u>TAINTED TURCO SOLUTION (mg/L)**</u>
Cobalt	<0.1	<0.1
Arsenic	<0.1	<0.1
Cadmium	<0.02	<0.02
Chromium	<0.05	<0.05
Copper	<0.02	<0.02
Iron	<0.02	1.09
Manganese	<0.02	<0.02
Nickel	<0.1	<0.1
Sodium	341	374
Magnesium	<0.05	<0.05
Potassium	<1.0	<1.0
Calcium	<0.5	<0.5
Barium	<0.02	<0.02
Mercury	<0.1	<0.1
Selenium	<0.2	<0.2
Silver	<0.02	<0.02
Lead	<0.1	<0.1
Zinc	<0.02	<0.02
Aluminum	<0.05	<0.05
Beryllium	<0.02	<0.02

* Unused solution of 20% Turco 3878 LF-NC in DI water.

** Solution (20% Turco 3878 LF-NC in DI water) from which discolored panels were removed.

TABLE XXVIII AC ENGINEERING HP9-4-30 STEEL PANEL INFORMATION

- Steel purchased by Republic Engineered Steel form Air Melt Heat
- Two billets purchased, approximately 4,040 lbs. each. Simultaneous heat treatment of the two billets provided by H & H Heat Treating, Inc., heat number 3844507. All panels received had this heat number painted on them. Heat treatment process was as follows:
 Normalize: 1650°F 1.0 Hr. Air Cool to Room Temp.
 Temper: 1150°F 12.0 Hrs. Air Cool.
 Hardness: Actual=HB 341. Required=HB 341 Maximum. Inspected 1 piece.
- Friend Metal purchased billets from Republic Engineered Steel. Our 30 panels were cut from a single billet, but unknown which of two.
- Analysis of steel metallurgy (Republic Engineered Steel) gave the following results:

<u>ELEMENT</u>	<u>SPECIFICATION</u> <u>AMS-6526C</u> <u>(ASRM 43003)</u>	<u>ACE PANEL</u>
Ni	7.4-8.0	7.88
Co	4.45-4.75	4.72
C	0.31-0.34	0.34
Mn	0.10-0.35	0.27
P	0.01 max.	0.007
S	0.003 max.	0.001
Si	0.15 max.	0.06
Cu	0.35 max.	0.16
Cr	0.90-1.10	1.06
Mo	0.90-1.10	1.02
V	0.06-0.12	0.108
O	0.004 max.	-
N	0.003 max.	-
Al	Report	-
Sb	Report	-
As	Report	-
Pb	Report	-
Sn	Report	-
Zn	Report	-

- Purchase and processing information based on phone conversations with personnel at Friend Metal and Republic Engineered Steels.

TABLE XXIX
BRINELL HARDNESS OF ACE HP9-4-30 PANELS

PANEL NUMBER	BRINELL HARDNESS	DISCOLORED
1	344-353	Y
2	525-543	-
3	301-371 (warped)	-
4	327-371	N
5	327-336	Y
6	271-319	-
7	353-353	Y
8	327-344	Y
9	336-344	Y
10	301-301	-
11	327-327	Y
12	344-344	Y
13	319-336	Y
14	344-362	N
15	-	-
16	-	-
17	-	-
18	319-327	Y
19	319-327	Y
20	-	-
21	319-336	Y
22	-	-
23	344-371	Y
24	-	-
25	-	-
26	344-353	Y
27	-	-
28	344-353	Y
29	344-353	Y
30	344-353	N

Two measurements made per panel with Rockwell C tester. Results converted to Brinell hardness (BHN).

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TABLE XXX
AEROJET AND AC ENGINEERING HP9-4-30 STEEL
PANEL ANALYSIS

ELEMENT	AEROJET PANELS		ACE PANELS				ASRM SPECIFICATION 43003
	X-RAY (MSFC)	ICP (SEAS)	X-RAY (MSFC)	X-RAY (REPUBLIC)	ICP (SEAS)	ICP (AMS)	
COPPER	ND	0.09	N.D.	0.16	0.12	0.14	0.35 MAX.
CHROMIUM	0.95-1.06	0.93	1.14	1.06	0.92	1.06	0.9-1.10
NICKEL	7.6-7.72	7.14	8.32-8.34	7.88	6.89	7.89	7.4-8.0
IRON	--	78.8	--	--	67.9	--	--
SULFUR	N.A.	<0.01	N.A.	0.001	<0.01	0.001	0.003 MAX.
PHOSPHORUS	0.006-0.01	<0.01	0.006	0.007	<0.01	0.007	0.01 MAX.
SILICON	0.06-0.1	<0.01	0.06-0.07	0.06	<0.01	0.08	0.15 MAX.
MANGANESE	0.22	0.28	0.22	0.27	0.26	0.28	0.1-0.35
COBALT	4.62-4.72	4.33	5.25-5.27	4.72	4.13	4.75	4.45-4.75
MOLYBDNEUM	0.89-1.02	1.72	1.1-1.12	1.02	1.79	1.0	0.9-1.10
VANADIUM	0.09-0.1	0.06	0.14-0.15	0.108	0.09	0.11	0.06-0.12

X-RAY ANALYSES PERFORMED AT MARSHALL SPACE FLIGHT CENTER (MSFC) AND REPUBLIC ENGINEERED STEEL.

ICP (INDUCTIVE COUPLED PLASMA) ANALYSES PERFORMED AT SOUTHEASTERN ANALYTICAL SERVICES (SEAS) AND ACCURATE METALLURGICAL SERVICES, INC. (AMS) --CUSTOMER FRIEND METALS CO., INC..

ABOVE ANALYSES PERFORMED IN AN EFFORT TO DETERMINE CAUSE OF AC ENGINEERING HP9-4-30 STEEL PANELS DISCOLORING DURING CLEANING WITH TURCO 3878 LF-NC.

TABLE XXXI
HP9-4-30 STEEL
EPDM INSULATION AND EPOXY BOND DATA
AFTER VARIOUS TURCO CLEANING RESPONSES

<u>PANEL</u>	<u>SURFACE</u> (see note below)	<u>INSULATION</u> <u>HARDNESS</u> <u>SHORE A/</u> <u>EPOXY*</u>	<u>MAX. PEEL</u> <u>LOAD</u> (lbs)** (avg. of 7 specimens)	<u>TENSILE</u> <u>ADHESION</u> (psi)*** (avg. of 7 specimens w/ EPDM, avg. of 10 w/ epoxy)	<u>FAILURE</u> <u>MODE****</u>
HP2	CLEAN (1)	EPOXY	N/A	225.2	80% EPOXY/ METAL 20% EPOXY
HP4	CLEAN (1)	80.9	152.7	449.3	INSULATION
HP30	CLEAN (1)	82.1	138.4	452.5	INSULATION
HP21	DARKENED (2)	EPOXY	N/A	676.7	50% EPOXY/ METAL 50% EPOXY
HP5	DARKENED (2)	81.0	166.3	473.2	INSULATION
HP29	DARKENED (2)	79.9	148.6	471.0	INSULATION
HP19	DARKENED BLASTED (3)	EPOXY	N/A	1023	100% EPOXY
HP7	DARKENED BLASTED (3)	81.7	154.0	489.0	INSULATION
HP13	DARKENED BLASTED (3)	83.1	154.7	475.9	INSULATION

NOTES:

* EA934.NA epoxy, 30 mil bondline, 1hr. cure under pressure, at 200°F.

**Peel test angle=45°, crosshead speed=2 in/min.

***Insulation tensile test pulled at 0.5 in/min crosshead speed. Epoxy tensile test pulled at 0.05 in/min crosshead speed.

****All insulation tensile buttons exhibited 100% cohesive insulation failure, and all peel specimens failed along the insulation/scrim interface. Epoxy tensile adhesion specimens exhibited mixed failure modes.

- 1) Panels did not discolor after repeated cleaning with Turco 3878 LF-NC.
- 2) Panels became discolored during cleaning with Turco 3878 LF-NC.
- 3) Panels became discolored during cleaning with Turco 3878 LF-NC, and were then zirconium silicate blasted to remove discoloration.

TABLE XXXII: HP9-4-30 OSEE SUMMARY¹

TAGUCHI RUN 1: 70°F/40%RH/4HR + 75°F/55%RH/24HR

PANEL	INITIAL OSEE/STD.DEV (cV)	POST EXPOSURE OSEE/STD. DEV (cV)	DELTA OSEE (cV)	CONTAMINANT TYPE/LEVEL MG/FT ²	FINAL OSEE (cV)/STD. DEV. Z
HP22	570/36	516/46	54	HD-2/25	189/10
HP27	575/22	553/22	22	HD-2/250	182/12
HP10	627/36	567/33	60	KAYDOL/20	59/44
HP23	583/37	505/33	78	KAYDOL/170	0/1
HP25	543/37	472/32	71	SILICONE/5	269/69
HP20	568/29	502/19	66	SILICONE/15	40/30
HP24	575/46	501/36	74	NA	NA
AVERAGE	577/35	516/32	61		

TAGUCHI RUN 2: 70°F/40%RH/48HR + 75°F/55%RH/24HR

PANEL	INITIAL OSEE/STD.DEV (cV)	POST EXPOSURE OSEE/STD. DEV (cV)	DELTA OSEE (cV)	CONTAMINANT TYPE/LEVEL MG/FT ²	FINAL OSEE (cV)/STD. DEV.
HP11	583/59	503/23	80	HD-2/17	212/35
HP1	632/58	542/27	90	HD-2/220	195/5
HP16	646/65	547/22	99	KAYDOL/20	209/100
HP7	575/31	497/26	78	KAYDOL/180	0/1
HP12	529/23	503/29	26	SILICONE/7	273/51
HP21	514/26	542/25	+28	SILICONE/15	137/54
HP13	484/47	498/49	+14	NA	NA
AVERAGE	566/48	518/32	47		

¹Contaminants were dissolved in methyl chloroform (5 mg/ml), then sprayed onto the panels using a Graco air brush (model G1265, series B) pressurized to 40 psi with nitrogen. Coating level was determined by measuring the weight change of aluminum witness foils sprayed along with the panels.

TABLE XXXII, CONTINUED

TAGUCHI RUN 3: 70°F/75%RH/4HR + 75°F/55%RH/24HR

PANEL	INITIAL OSEE/STD.DEV (cV)	POST EXPOSURE OSEE/STD. DEV (cV)	DELTA OSEE (cV)	CONTAMINANT TYPE/LEVEL MG/FT ²	FINAL OSEE (cV)/STD. DEV.
HP10	521/24	572/22	+51	HD-2/27	131/8
HP7	466/23	517/22	+51	HD-2/210	153/8
HP12	482/23	497/21	+15	KAYDOL/29	36/11
HP16	494/25	505/20	+11	KAYDOL/218	21/5
HP24	459/31	537/30	+78	SILICONE/2	294/23
HP27	461/41	501/66	+40	SILICONE/17	45/11
HP13	447/28	528/14	+81	NA	NA
AVERAGE	476/28	523/28	+47 ²		

2- Ni standard averaged 909 cV when initial measurements were made, and 980 cV when post exposure measurements were made

TAGUCHI RUN 4: 70°F/75%RH/48HR + 75°F/55%RH/24HR

PANEL	INITIAL OSEE/STD.DEV (cV)	POST EXPOSURE OSEE/STD. DEV (cV)	DELTA OSEE (cV)	CONTAMINANT TYPE/LEVEL MG/FT ²	FINAL OSEE (cV)/STD. DEV.
HP30	468/34	472/30	+4	HD-2/21	147/18
HP4	561/42	496/32	65	HD-2/195	170/13
HP29	485/32	490/32	+5	KAYDOL/28	214/84
HP5	521/28	470/19	51	KAYDOL/195	0/1
HP19	533/28	492/17	41	SILICONE/4	330/8
HP17	547/32	510/23	37	SILICONE/17	27/21
HP14	493/31	504/17	+11	NA	NA
AVERAGE	515/32	490/24	25		

TABLE XXXII, CONTINUED

TAGUCHI RUN 6: 80°F/40%RH/48HR + 75°F/55%RH/24HR

PANEL	INITIAL OSEE/STD.DEV (cV)	POST EXPOSURE OSEE/STD. DEV (cV)	DELTA OSEE (cV)	CONTAMINANT TYPE/LEVEL MG/FT ²	FINAL OSEE (cV)/STD. DEV.
HP1	505/30	445/20	60	HD-2/27	138/7
HP23	489/26	472/22	17	HD-2/210	161/5
HP22	483/24	464/21	29	KAYDOL/29	32/8
HP21	506/20	498/20	8	KAYDOL/218	21/4
HP25	483/27	461/27	2	SILICONE/2	324/28
HP20	488/32	447/27	21	SILICONE/17	50/18
HP11	502/29	444/15	58	NA	NA
AVERAGE	489/27	462/22	28		

TABLE XXXIII: HP9-4-30/EPDM BOND TEST SUMMARY¹

TAGUCHI RUN 1: 70°F/40%RH/4HR + 75°F/55%RH/24HR

PANEL	INSULATION HARDNESS, SHORE "A"	COATING TYPE/LEVEL, MG/FT ²	AVG. PEEL ADHESION, PLI/FAILURE	AVG. TENSILE ADHESION, PSI/FAILURE
HP22	77	HD-2/25	103/100% I	252/100% I
HP27	82	HD-2/250	103/95% I, 5% CLA	324/100% I
HP10	84	KAYDOL/20	95/100% I	365/100% I
HP23	84	KAYDOL/170	36/100% I	371/100% I
HP25	82	SILICONE/5	101/100% I	316/100% I
HP20	81	SILICONE/15	71/100% I	371/100% I
HP24	83	NA	42/100% I	290/100% I

TAGUCHI RUN 2: 70°F/40%RH/48HR + 75°F/55%RH/24HR

PANEL	INSULATION HARDNESS, SHORE "A"	COATING TYPE/LEVEL, MG/FT ²	AVG. PEEL ADHESION, PLI/FAILURE	AVG. TENSILE ADHESION, PSI/FAILURE
HP11	82	HD-2/17	118/100% I	296/100% I
HP1	82	HD-2/220	103/95% I, 5% CLA	300/100% I
HP16	76	KAYDOL/20	119/100% I	265/100% I
HP7	85	KAYDOL/180	107/100% I	406/100% I
HP12	83	SILICONE/7	103/100% I	310/100% I
HP21	82	SILICONE/15	109/100% I	247/100% I
HP13	83	NA	106/100% I	349/100% I

¹Peel adhesion tested at 45 degrees, 2 inches per minute. Tensile adhesion tested at .5 inches per minute. Reported peel strengths represent the average of 5 specimens tested, and the tensile strengths are the average of six specimens.

Contaminants were dissolved in methyl chloroform (5 mg/ml solutions), then sprayed onto the panels using a Graco air brush (model G1265, series B) pressurized to 40 psi with nitrogen. Coating level was determined by measuring the weight change of aluminum witness foils sprayed along with the panels.

I=cohesive insulation failure; CLA=Chemlok to insulation adhesive failure
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TABLE XXXIII, CONTINUED

TAGUCHI RUN 3: 70°F/75%RH/4HR + 75°F/55%RH/24HR

<u>PANEL</u>	<u>INSULATION HARDNESS, SHORE "A"</u>	<u>COATING TYPE/LEVEL, MG/FT²</u>	<u>AVG. PEEL ADHESION, PLI/FAILURE</u>	<u>AVG. TENSILE ADHESION, PSI/FAILURE</u>
HP10	86	HD-2/27	99/100% I	460/100% I
HP7	84	HD-2/210	94/100% I	459/50% I, 50% CL/S
HP12	83	KAYDOL/29	113/100% I	451/100% I
HP16	83	KAYDOL/218	84/100% I	454/100% I
HP24	82	SILICONE/2	106/100% I	453/100% I
HP27	83	SILICONE/17	88/100% I	468/100% I
HP13	84	NA	103/100% I	463/100% I

TAGUCHI RUN 4: 70°F/75%RH/48HR + 75°F/55%RH/24HR

<u>PANEL</u>	<u>INSULATION HARDNESS, SHORE "A"</u>	<u>COATING TYPE/LEVEL, MG/FT²</u>	<u>AVG. PEEL ADHESION, PLI/FAILURE</u>	<u>AVG. TENSILE ADHESION, PSI/FAILURE</u>
HP30	82	HD-2/21	NO DATA ²	319/100% I
HP4	81	HD-2/196	65/100% I	387/100% I
HP29	84	KAYDOL/28	104/100% I	310/100% I
HP5	82	KAYDOL/195	87/100% I	368/100% I
HP19	82	SILICONE/4	110/100% I	298/100% I
HP17	85	SILICONE/17	119/100% I	352/100% I
HP14	82	NA	110/100% I	349/100% I

2-Scrim inadvertently left out of specimens, therefore tabs broke during testing

TABLE XXXIII, CONTINUED

TAGUCHI RUN 6: 80°F/40%RH/48HR + 75°F/55%RH/24HR

<u>PANEL</u>	<u>INSULATION HARDNESS, SHORE "A"</u>	<u>COATING TYPE/LEVEL, MG/FT²</u>	<u>AVG. PEEL ADHESION, PLI/FAILURE</u>	<u>AVG. TENSILE ADHESION, PSI/FAILURE</u>
HP1	83	HD-2/27	111/100% I	454/100% I
HP23	85	HD-2/210	105/95% I, 5% CL1	447/100% I
HP22	87	KAYDOL/29	110/100% I	466/100% I
HP21	84	KAYDOL/218	102/100% I	432/100% I
HP25	83	SILICONE/2	113/100% I	462/100% I
HP20	81	SILICONE/17	106/100% I	440/100% I
HP11	86	NA	107/100% I	465/100% I

TABLE XXXIV

EPDM 44010 MECHANICAL PROPERTY SUMMARY FOR CURED INSULATION

<u>TEST DATE/LOCATION</u>	<u>AVG. HARDNESS, SHORE "A"</u>	<u>ORIENTATION</u>	<u>AVG. STRESS AT MAX. LOAD, PSI</u>	<u>AVG. MAX. PERCENT STRAIN, %</u>
4/30/93-RM ENGINEERED PRODUCTS	92	PARALLEL TO FIBER	2457	14
4/30/93-RM ENGINEERED PRODUCTS	92	PERPENDICULAR TO FIBER	1041	52
6/8/93-MSFC	82	PARALLEL TO FIBER	2290	28
6/8/93-MSFC	82	PERPENDICULAR TO FIBER	973	89
10/31/93-MSFC	82	PARALLEL TO FIBER	2188	19
10/31/93-MSFC	82	PERPENDICULAR TO FIBER	884	78

• SPECIFICATION REQUIREMENTS FOR CURED EPDM 44010 ARE AS FOLLOWS:

- STRESS AT MAX LOAD PARALLEL TO FIBER ≥ 1000 PSI
- MAX PERCENT STRAIN PARALLEL TO FIBER $\geq 5\%$
- STRESS AT MAX LOAD PERPENDICULAR TO FIBER ≥ 500 PSI
- MAX PERCENT STRAIN PERPENDICULAR TO FIBER $\geq 5\%$
- HARDNESS, SHORE "A" ≥ 80

• MSFC SHORE "A" HARDNESS/MECHANICAL PROPERTY DATA REPRESENTS THE AVERAGE RESULTS OF FIVE JANNAF TYPE "C" TENSILE DOGBONES TESTED. RM ENGINEERED PRODUCTS DATA WAS THE AVERAGE OF THREE TENSILE DOGBONES.

• TEST RATE WAS 20 INCHES/MINUTE CROSSHEAD SPEED.